Diffraction Control Unit
PW 1710/00

Operation Manual

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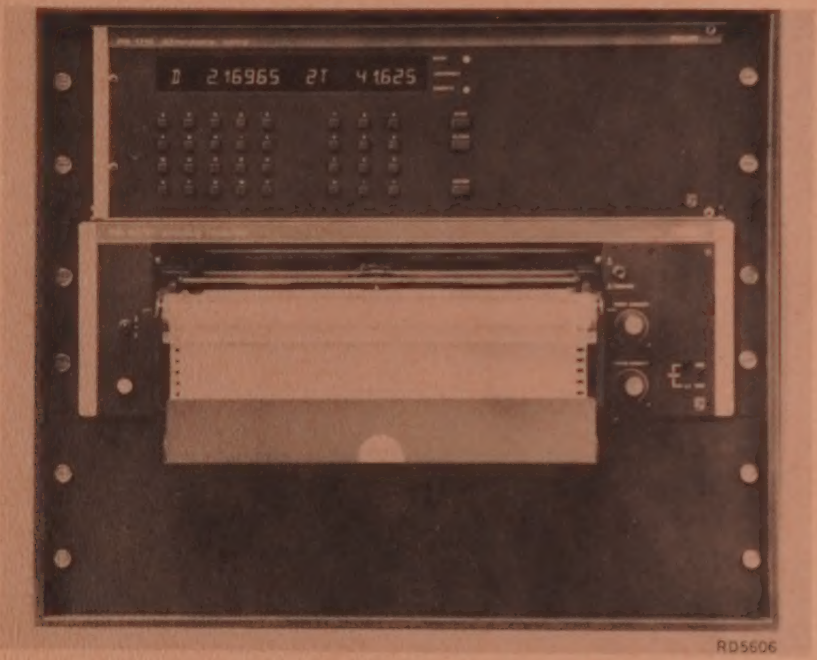
850920



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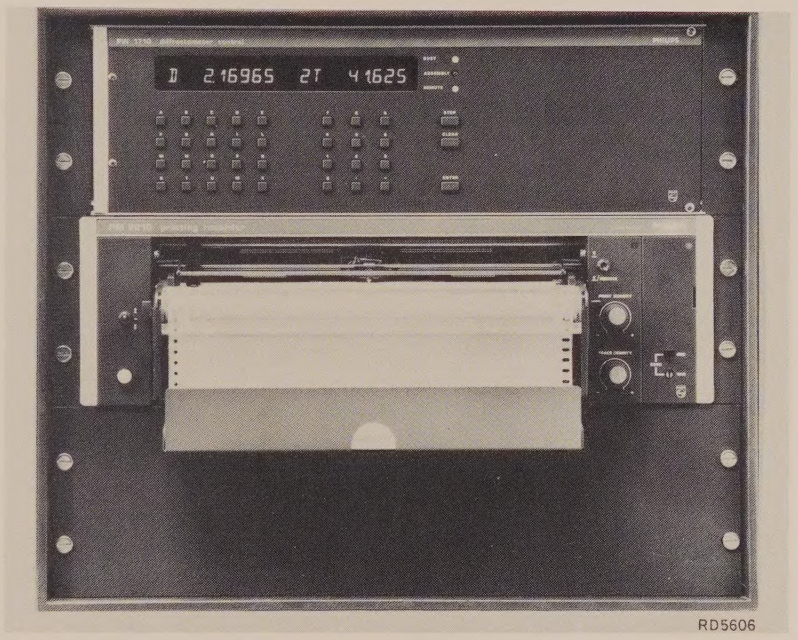

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EDITION NOTICE 2nd Edition

This is the second edition of this publication. It is intended for use with a variety of configuration options for the PW1710 X-ray Diffractometer,

PW1710 Operation Manual.

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1.1 INTRODUCTION

The Philips automated powder diffractometry system consists of several units and options. This manual descibes the operation of the PW1710 diffractometer control, one of the units of the system. The main functions of the PW1710 are:

- Control of the goniometer, sample changer, spinner and high voltage setting of the detector.
- Pulse height selection and counting of the x-ray photons of interest.
- Calculation and printing of d-values on diffractograms and the terminal.
- Evaluation of peak intensities and peak areas.
- Calculation of phase concentrations with user-selected algorithms.
- Input of commands, printout and display of data, status signals and error messages.
- Execution of commands and user-made programs.

For information on the installation and operation of other units and options for the automated powder diffractometry system refer to the respective manuals, as delivered. A brief description of units and options available is also provided in this manual to help you to understand the overall operation of the system.

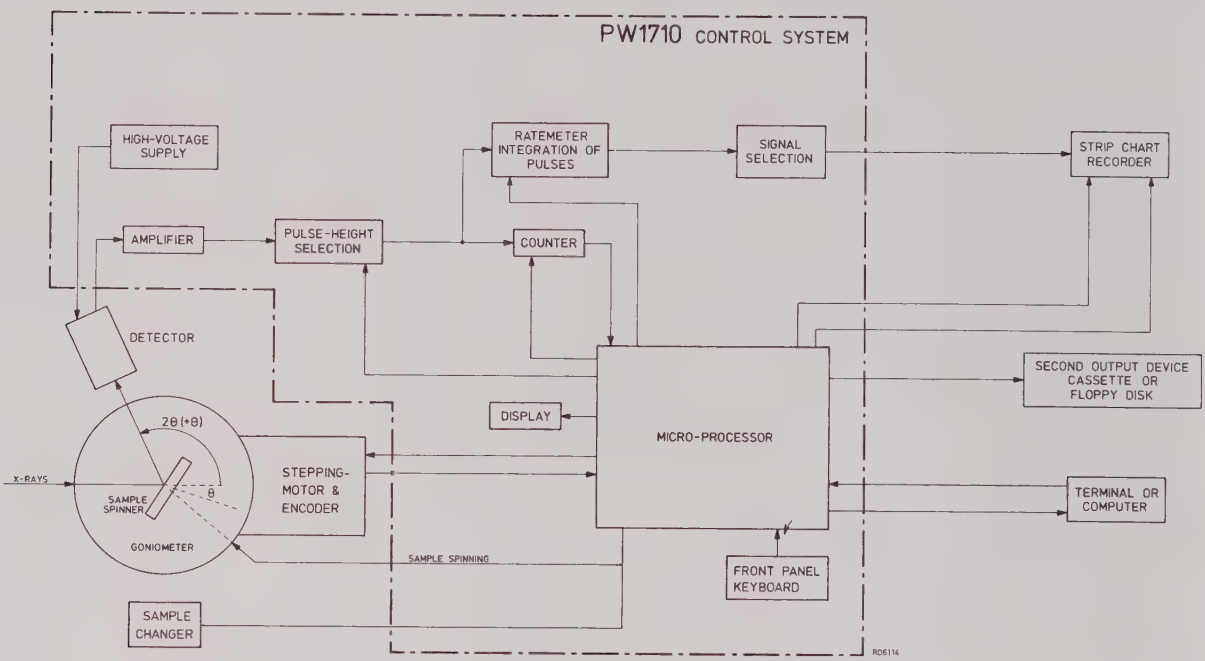

Figure 1.1 The Main Functions of the PW1710

1.2 DESCRIPTION OF THE PW1710 DIFFRACTOMETER CONTROL

1.2.1 FRONT PANEL

The PW1710 front panel consists of:

- A 32-key alphanumeric keyboard for input of measurement parameters and commands when no separate input/output terminal is used with the system.
- A 20-digit alphanumeric readout to give a variety of information including commands, parameters, error signals, and available memory space or output of results.
- System status indicators to show when the system is BUSY, in the ASSEMBLY mode, or in the REMOTE mode communicating with a terminal or computer.
- Command push-buttons to carry out control functions.
- A hole allowing access to the RESET control.

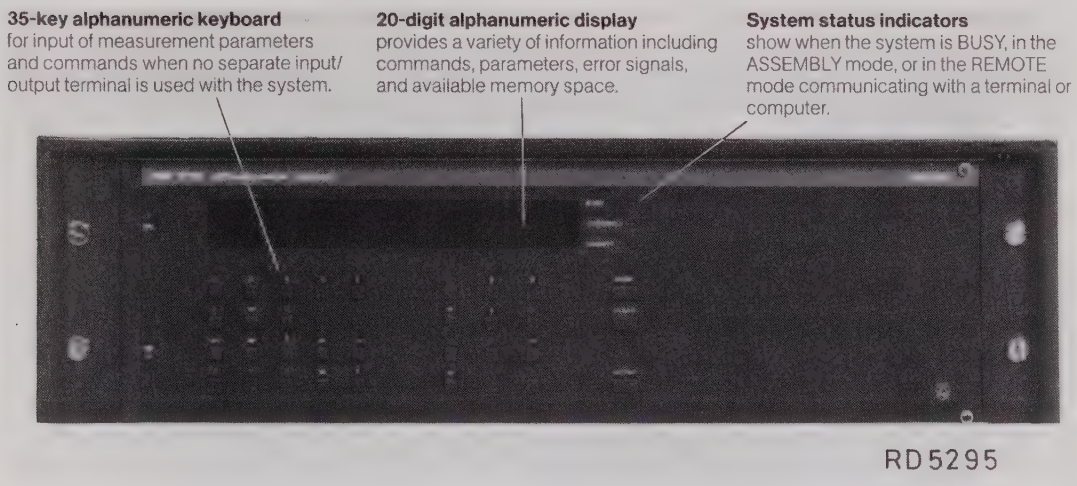
35-key alphanumeric keyboard
for input of measurement parameters and commands when no separate input/output terminal is used with the system.
20-digit alphanumeric display
provides a variety of information including commands, parameters, error signals, and available memory space.
System status indicators
show when the system is BUSY, in the ASSEMBLY mode, or in the REMOTE mode communicating with a terminal or computer.
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Figure 1.2 PW1710 Diffractometer Control Front Panel

The PW1710 is the Control Unit of a diffractometry system. All the data and control signals for the system are routed through the PW1710 microprocessor, which in turn receives commands from an operator's terminal, a computer, or the PW1710 front panel. Remote commands can also be entered via the front panel. A sequence of commands can be stored in memory, under a program number, and carried out using a single start command.

Part of the PW1710 memory can be reserved for the storage of measuring data. Calculator functions are also available to enable simple calculations. Output format can also be specified, for particular jobs.

The PW1710 front panel incorporates an alphanumeric keyboard, an alphanumeric display, command push-buttons (STOP, CLEAR and ENTER) and system status indicators. On systems with no separate operator's terminal all command input and result presentation is controlled from the front panel. On systems fitted with a separate operator's terminal the front panel can still be used to display parameters and values and carry out local control when required.

The most common configuration of the system is with an operator's terminal and for this reason the manual concentrates on this layout. A separate chapter is devoted to system operation without an operator's terminal. Most system control remains the same but, where there are differences they are highlighted and explained. When operating in from the front panel the letter "U" must be substiruted for "V" during command entry, as there is no "V" on the front panel.

1.2.2 SECONDARY CONTROL PANEL

The secondary control panel carries the following controls to set the detectors and certain functions which only need to be set occasionally:

- A selector switch for the x-ray tube anode.
- A switch to set the detector pulse attenuation.
- A push-button to reset the system.
- Slide switches to define the system configuration.
- A push-button to switch off the pulse shift correction.

Once the system has been set up manually for a specific wavelength, changeover to a tube with a different anode only requires that the selector switch be set to the corresponding element name (Cu, Co, Fe, Cr or Mo). The attenuation is automatically adjusted to suit the new anode type.

The secondary control panel settings do not need to be changed during normal operations. If another x-ray tube is fitted, or the system configuration is changed, the switches must be set to the appropriate positions (refer to the Service Manual Section 2.8).

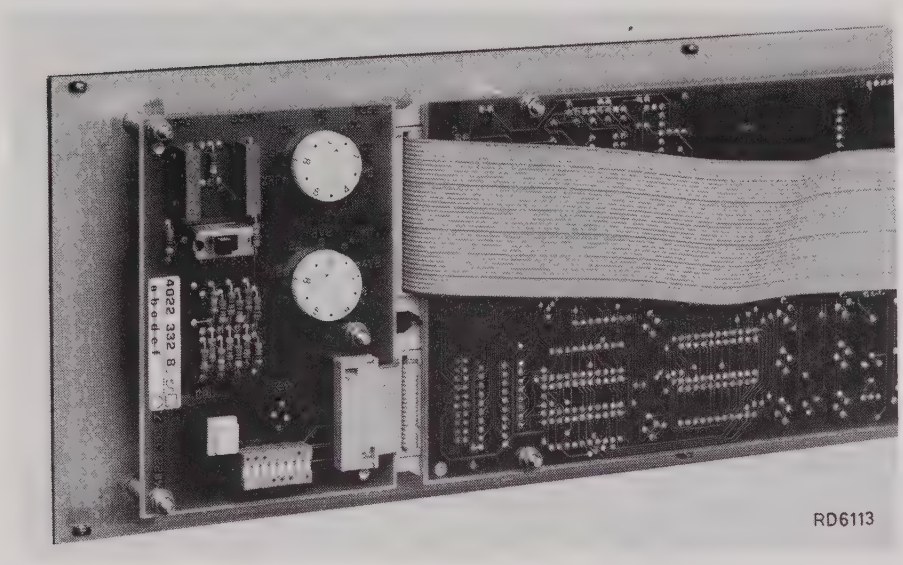


Figure 1.3 PW1710 Diffractometer Control Secondary Control Panel

1.2.3 OPERATOR´S TERMINAL

The system can be fitted with a variety of different terminals for use by the operator. Teletypewriters or visual display units, VDUs, can be used sas operator´s terminals, and it is also possible to fit a printer as a secondary output terminal if required. This provides a very useful facility when used in conjunction with a VDU as it allows use of the printer to produce hard copy of program listings, results and so on.

Operation of the system is similar, whether operating in FRONT PANEL or REMOTE mode, although there are minor variations, necessitated by different output and display facilities, together with the different controls on the front panel.

A further facility allows entry into a special MANUAL mode from REMOTE or FRONT PANEL mode. The MANUAL mode allows entry of special abbreviated versions of each of the commands, from the front panel. Each command requires a shorter entry, but it is not possible to run programs.

1.2.4 MICROPROCESSOR AND MEMORY

The main parts of the PW1710 Diffractometer Control are a microprocessor and a memory (see Figure 1.4). The microprocessor controls the handling of commands and data. The memory consists of:

- An output buffer.
- An accumulator.
- Registers.
- A calculator.

The memory will also store data and commands from programs entered by the user, for immediate or later use. The storage capacity of the memory is limited, therefore a digital cassette recorder is often used to provide additional storage facilities. Application programs for the diffraction system can then be stored on cassettes, and an unlimited number of cassettes can be used.

The output buffer is used to make the processed information available, in a suitable form for output to a computer, printing recorder or hardcopier. The accumulator is a part of the memory where the microprocessor stores and recalls data for further processing. Registers store data until recalled or overwritten by the user (user commands).

The calculator carries out measurement results (data) processing. Commands to the microprocessor can be given from the front panel keyboard or from a terminal in REMOTE mode. Measurement intensities, the actual 2θ diffractometer angle and the memory capacity of the registers, are given on the front panel readout.

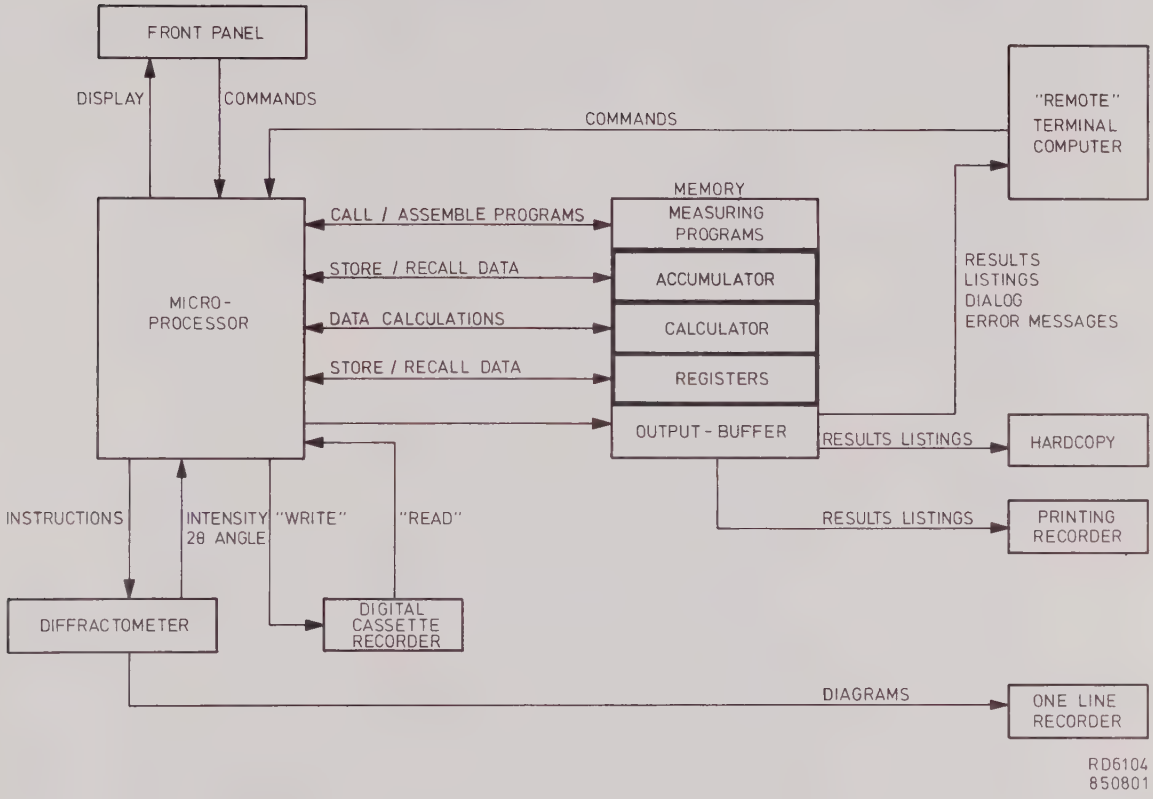


Figure 1.4 PW1710 Diffractometer Control

1.3 ABBREVIATIONS USED IN THIS MANUAL

2D	Second derivative file
A2	Results of K-alpha$_2$ stripping (data file)
AMORPH	Command - Locate "AMORPHOUS" peaks
AMS	Additive Match Score of a reference pattern explaining the experimental diffraction complementary to other patterns.
APD	Automated Powder Diffraction
APR	April
AUG	August
BK	Background
<CR>	(Carriage) Return
CRT	Video terminal (Cathode Ray Tube)
D1 - D9	Results from Subtract (data file)
D01 - D99	Parameter files
DAT	Parameter file
DCL	Digital command language
DD/D	Delta d over d (d-spacing)
DEC	December
DEC*	Digital Equipment Corporation*
DEF	Default
DEL	Delete (a) file(s)
dev	device
DI	d-spacings and Intensities file
DIR	(display) Directory of files
DM	Data Mark (in a match score tabel) as found on the PDF cards
E	Exit (from the APD program = return to RSX operinting systems)
EQDE	Equivalent Displacement Error of the sample in micrometres. Calculated from the average deviation of the measured peak position from the reference peak position.
FCS	File Control Services
FEB	February
FEPF	Frequently Encountered Phases (a sub-file of powder diffraction data base)
G	Go
g	group of users in UFD
HEL	Hello (to the system): Log in
HP	Hewlett Packard
I%	Intensity factor indicating percentage of scaling used
JAN	January
JB	Batch file
JCPDS	International centre for diffraction data (formerly the Joint Committee on Powder Diffraction Standards)
JUL	July
JUN	June
LO	LOG out
LOG	LOG (in or out)
m	member of UFD
MAR	March
MAY	May

MCR	Monitor Console Routine
MLMS	Maximum Likelihood Match Score of a reference pattern explaining the experimental diffraction pattern
MS	Match score table/file, result of phase identification (IDENTIFY)
N	No
NBS	National Bureau of Standards (USA) high-quality reference patterns
NOV	November
OCT	October
PDF	Powder Diffraction File
PDP*	Programmable Data Processor
PK	Peak file, data file from which the backgound is removed
Q	Quit
Q1	Quantitative Intensities file
RD	Raw data file, result of Data collection
RSX*	Resource Sharing Executive (a DEC Operating system for PDP11 computers)
SANDMAN	Search AND MAtch on Nova (name for phase identification program)
SEP	September
SF	Sub file, for Restriction in Identify (SANDMAN)
SM	Smoothed data file
SPR	Software Performance Report
UFD	User File Directory
UIC	User Identification Code
VDU	Video terminal (Video Display Unit)
VT*	Video Terminal (VT100, VT125 and so on
WK	(temporary) work file, used during data collection
Y	Yes

* DEC, PDP, RSX, and VT are trade marks of Digital Equipment Corporation

1.4 HOW TO USE THE PW1710

You can use two different systems to learn to use the PW1710 Diffractometer Control system. They are:

- By trial and error.
- By following the commands given in this manual.

The first method looks easy to people who know the trade and have a synopsis of the commands to hand. Experience however has shown that using the second method is better. The time wasted in trying to understand strange results could be better spent reading the instructions. For this reason we strongly advise you to take time to read this manual, as it is based on our own experience.

This manual is designed as a guide through from the beginning to complete use of all the facilities provided by the system. It starts with the procedure used to switch on and tune the instrument to get the best results. It then explains how to measure, how to get the results out, and how to program the system.

Do not be afraid of programming. Programming means instructing the machine to perform procedures. The first step in programming is to decide what is to be done. This manual details the procedure to make a suitable program once the requirements have been defined. When programs have been made they can be stored on the memory in the instrument. They can be recalled later on by entry of a simple number.

The initial steps to learn how to use the instrument are:

- Learning the commands.
- Making simple programs to carry out some of the tasks needed for a job.
- Making complete programs to carry out a job and using them.

1.5 SWITCHING ON

1.5.1 HOW TO SWITCH ON

The PW1710 Diffractometer Control can be:

- Built-in in a PW1730 generator.
- In a separate cabinet.

Use the following procedure to switch the system on:

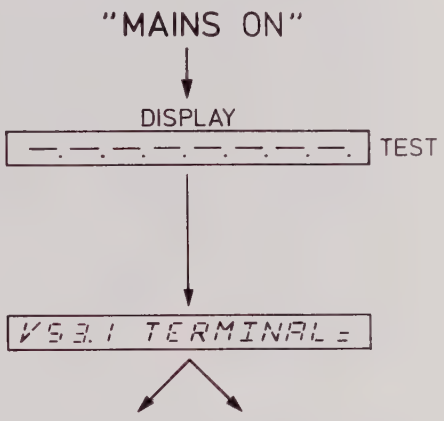
- Switch the system power supply ON. The procedure to do this depends on the system configuration.
- Switch the PW1710 ON.

1.5.2 AFTER SWITCHING ON

When the switch on procedure has been completed a series of points and lines will be displayed on the readout, and on the terminal when there is one in use. These characters will be removed after a few seconds, to indicate that a successful system memory test has been carried out, and the switch on procedure continues. If the characters "C" or "X" remain displayed call a service engineer and do not try to carry on.

The readout displays the message:

VS x.x TERMINAL =



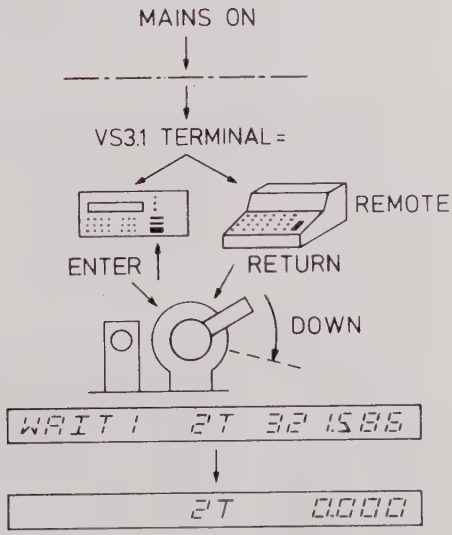
The same message is sent to the operator's terminal, if available, and is shown there. The BUSY lamp stays on and the system waits for an operator action.

This message is used to perform a dual function:

- VS x.x indicates that version x.x of the operating software is fitted to the PW1710.
- TERMINAL = is a prompt for selection of operation from the front panel push-buttons or from a remote terminal.

Select front panel operation by pressing the ENTER push-button on the front panel. Select operation from a remote terminal by pressing the key marked RETURN or ENTER on the on the operator's terminal keyboard. This key is identified by <CR> throughout this manual. If operating in FRONT PANEL mode <CR> refers to the push-button marked ENTER on the front panel. The BUSY lamp on the front panel is always lit after selection of the operational mode and the REMOTE lamp also lights if REMOTE mode is selected.

FRONT PANEL mode means that control of the system is from the PW1710 front panel. REMOTE mode means that control of the system is from the remote terminal, or computer. If control is from a remote computer it will translate the commands given via the keyboard. After selection of the mode a start program is automatically loaded. This start program is referred to as program "0".



After mode selection program "0" runs and the goniometer moves downwards. The message:

WAIT 1

is displayed on the left of the readout, to indicate that the system is waiting for the goniometer. The 2θ angle is displayed on the right together with some running figures. The goniometer moves down to zero degrees, calibrates, and then moves up to the angle specified in the program. The angle specified in program "0" as supplied is five degrees, although this may have been changed by other operators if the system has been installed for a while. When the goniometer reaches the angle specified the message on the left is removed and the 2θ angle is displayed on the readout.

C=?
WAITING FOR AN INSTRUCTION/COMMAND

The system is now ready for use and instructions (commands) can be entered. The system displays the prompt:

C =

and the BUSY lamp on the front panel is out to indicate readiness for use.

INSTRUCTION
(COMMAND)

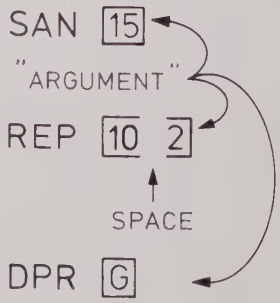
1.6 INSTRUCTIONS (COMMANDS)

Commands are coded instructions which are used to control the machine. These commands can be given in two different ways:

- Entering each command step by step for direct control.
- Combining commands into a program using the program assembling facility.

A program is made up of a series of commands. Each program on the PW1710 is identified by a number unique to the program. There are 256 numbers (0 to 255) available for use on the PW1710. Program "0" is reserved for use as the starting program. Most of the commands which you can use on the system can be included in programs. The commands which cannot be included in programs are those used to make them, to carry out certain printing instructions, to carry out tests and so on.

An command is made up of three or four letters. These may be followed by one or more characters to control system operation, and are always ended with a <CR> in REMOTE mode and by pressing the ENTER push-button in FRONT PANEL mode. Instructions are often called commands, and accompanying characters are called arguments. Arguments can be numeric values, certain letters or combinations of numeric and alphabetic characters.



All the commands are designed in mnemonic form. This means that the letters used to make up the command are an abbreviation of the function performed. As you get used to the system you will start to see the relationship between commands and their functions. All non-programmable commands are identified in the command summary.

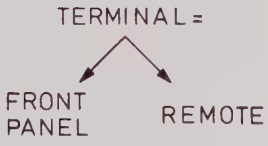
The following section introduces some of the basic commands most frequently used to operate the system. It is important to remember that there should never be any spaces in front of commands, although spaces can be placed between commands and their arguments. When using commands requiring more than one argument, the multiple arguments should always be separated using a comma or space in REMOTE mode. In FRONT PANEL mode use two stops, "..", to separate arguments. Typical command entries are:

SPACE = "SPACE BAR" (REMOTE)

SPACE = .. (FRONT PANEL)

- RON means set the to recorder ON.
- SAN 15 means set the 2θ goniometer angle.
- REP 10 2 means repeat program "10" twice.
- DPR G means display on the right of the screen or readout, and G refers to the 2θ goniometer angle.

When operating in FRONT PANEL mode all the commands must be given via the front panel. When operating in REMOTE mode all the commands must be given via an operator's terminal which can be:



- A teleprinter.
- A VDU.
- A computer where the commands can be given as abbreviations. The computer will translate the information into the normally used three-letter coded information (see the Survey of Commands).

When operating in the REMOTE mode the REMOTE lamp is lit and readout displays can be selected using the short commands incorporated in the system for use in MANUAL mode. These commands are detailed later on in this manual. In REMOTE mode the system can be reset by entry of:

//<CR>

on the keyboard. This returns the system to the switch on procedure. If the system is in MANUAL mode you can reset it by inserting the tip of a ball point pen in the hole on the front panel and pressing the RESET control.

Throughout this manual examples of simple programs are given, with comments in lower case explaining the function of each command. In some cases the commands have not been detailed in the text before being used but the explanations provide sufficient information to understand their function.

The PW1710 always presents the counts in counts per second, rather than total counts. This feature is an integral part of the software and must be considered when dealing with results. The system has been implemented in this manner for a number of reasons. It allows easy comparison between results without the need for correction factors when different measurement parameters have been used.

When using the system in front panel mode a special abbreviated form of command entry can be used. These abbreviated entries are detailed in the section on front panel o@peration. This special method of command entry is sometimes referred to as the MANUAL mode.

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2.1 INTRODUCTION

This chapter introduces the commands to be used after switching the PW1710 ON, and the commands to set the machine up. The instructions used to display information are also included here. The instructions are dealt with in the order that they will be required when starting to use the PW1710. Throughout the manual examples of short programs are given, to give an idea of what they look like; familiarity with them will help when starting to program. Where these examples occur, lower case text without full stops is used to explain them. Examples should not be confused with individual command explanations which are presented in a slightly different format.

The text describes the instructions to be used in REMOTE mode. When <ENTER> appears, in the FRONT PANEL mode, it refers to the ENTER push-button on the front panel. Incorrect command entry causes display of one of the following messages;

ILLEGAL ARGUMENT

or:

UNKNOWN COMMAND

Clear the error condition indicated by these messages by pressing <CR> in REMOTE mode, or the CLEAR push-button if in FRONT PANEL mode. The setting up instructions are grouped under the following headings:

- Parameters.
- Angle setting.
- Detector.
- Recorder.

2.2 INSTRUCTIONS FOR USE AFTER SWITCHING ON

The following instructions are normally used immediately after switching the PW1710 on. The first group are instructions to be used if a printing device is connected to the PW1710. Certain instructions which are used frequently have also been included here in order to introduce them. All the instructions in this section can be entered when the prompt:

C=

is displayed on the readout, and the terminal if in REMOTE mode. Selection of REMOTE or FRONT PANEL mode is detailed in the previous chapter.

HED Give the instruction HED<CR>. The system will respond by displaying:

00-00-00 00.00 PROGRAM 0

or a string of text and some values followed by the date, time and the number of the last program selected, for example:

ABCD XX FEC 22-11-82 17.40 PROGRAM 0

DAT Give the date after this command in the form dd mm yy as in the following example:

DAT 23 10 92<CR>

Enter each of the numbers that make up the date separated by spaces as follow:

- The day dd (or d).
- The month mm (or m).
- The year (minus 1900) separated by spaces.

TIM Enter the time after this command in the form hh mm, using the 24 hour clock as in the folowing example:

TIM 14 23<CR>

IDT This command is used to enter the text to be output when using the print heading instruction. Enter this instruction followed by up to 12 alphanumeric characters. This string of characters can include spaces, and is used to enter one of the following:

- A sample name.
- A sample code or series code.
- The operator´s name or text to identify the sample being tested.

2.3 SETTING UP INSTRUCTIONS

2.3.1 PARAMETERS

The basic function of the PW1710 is to act as a piece of measuring equipment. It should be tuned properly, before use, to ensure good results. It is also possible to tune major instructions in order to enhance operation of the system. Tuning is carried out by setting parameters. All these parameters must be given values before any operations can be carried out. Once values have been assigned to parameters they remain active until they are changed or the machine is switched OFF. Always check the values assigned to parameters before doing any measurements. Instructions used to specify the output device to be used to print the parameter values are also included in this section.

PAR This instruction is used to produce a list of all the parameters currently set on the machine. This listing can be output on any of the following:

- On the operator's terminal.
- On a second output device, if dual output has been specified.
- On the front panel display when operating in FRONT PANEL mode.
- On a printing recorder, if there is one fitted to the system, and it has been specified for output.

The following paragraphs introduce instructions to control the destination of the parameter listing produced by the print parameters command.

PON This instruction is used to enable the printhead on the recorder, if fitted. In other words it enables printing on the recorder chart.

POF This instruction is used to disable the recorder printhead.

DON This instruction is similar to the printhead enable command, and is used to specify dual output ON. It is used to specify output to two separate devices, when fitted.

DOF This instruction is used to switch dual output OFF.

DPL D This instruction is used to display the value of the lattice spacing, "d". This value is calculated from the 2θ position of the goniometer, as displayed on the readout. Calculation of the value is performed using Bragg's law.

$$2d \sin \theta = w$$

The expression "w", in the equation is the wavelength of the actual radiation line of the x-ray tube in use. This line is programmed using the set wavelength command. The current value for "w" can be found using the print parameters command. The wavelength is expressed in Angstrom units, with the lattice spacing "d" calculated and displayed for the current goniometer position. If the x-ray line wavelength is changed, the value "w" must be changed accordingly. Refer to the set the wavelength command.

DPR G This instruction is used to display the 2θ goniometer angle on the right hand side of the display.

PRP p This instruction is used to print the program specified by the argument "p". If no program number is specified as an argument all the programs are printed.

The following example demonstrates use of the print heading command in a program. This is a very useful instruction as it allows printing of program identification, together with the date and the elapsed time of the measurement. Several of the instructions in the example have not been explained yet, but their functions are detailed in the comments. It is assumed that the program specified for output has been installed on the system, and that the heading has been entered using the instructions detailed in this section. All the comments in lower case letters are explanatory, in all the examples, and do not appear on outputs. The example shown can be obtained, on display of the command entry prompt, by entry of the following:

PRP 60<CR>

```
HED             print the program heading
SAN 15.000      set the 2θ goniometer angle to 15°
ROF             recorder/ratemeter OFF (not used)
SPE 0.03        scan speed 0.03° 2θ / sec
INT 1.00        sampling interval time 1 second
CSP 10.000      make continuous scan from 15° to 15° + 10° with
                peak labelling
HED             print the program heading
```

The following is an example of the output when program "60", as listed earlier, is run. Note that the heading printed must have been set up using the give date, the give time and the give identity commands. The program name, DEMO, was entered using the give identity command.

```
DEMO  21-05-1985 10.34 PROGRAM 60
 15.229 5.8177 1360
 15.697 5.6533  923
 17.050 5.2002 1895
 17.871 4.9632 1057
 19.549 4.5407 1754
 19.993 4.4409 1535
 22.866 3.8890 2296
 24.814 3.5880  674
 DEMO  21-05-1985 10.34 PROGRAM 60
```

The following is an example of typical output obtained on entry of the print parameters command. The information in lower case is explanatory and is not output.

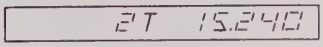
LWL 35 UPL 70	full-scale setting of ratemeter 2E5. set lower level at 35%, upper at 70%.
RFS 2E3	full-scale setting of ratemeter 2E3.
RTC .5 RSP 10	recorder time constant-5 second. recorder speed 10 mm / 2θ°.
SPE 0.10 INT 2.00	scan speed 0.10° 2 sampling interval time 2 seconds.
STP 0.050 MXN 10	step size 2θ 0.05°. number of measurement points on either side of angle.
MXS 0.010 MXT 1.00	step of 2θ 0.01° between measuring points. measurement 1 second.
BGT 3.00 BGM 2	background measurement 3 seconds. background 2.
BGA 0.500 WAV 1.54186	background offset 2θ 0.5°. wavelength 1.54180.
SOF RON DON POF	spinner off. recorder/ratemeter on. dual output on. printing recorder off.
ZERO2T -0.150	set the goniometer "ZERO"-position

2.3.2 ANGLE SETTING

The instructions detailed in this section are used to set the goniometer angle required. Before trying to use any of the instructions given in this section read all the text to ensure a full understanding of the effects of all these commands.

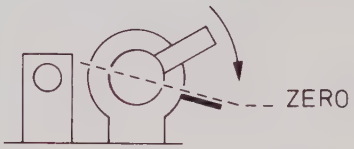
DPR G This instruction is used to output the 2θ goniometer angle. The value is displayed on the right hand side of the readout.

CAN a This instruction is used to correct the angular readout value. The angular position of the goniometer is given the 2θ angle "a". The readout is changed causing the scale to shift (offset) by a fixed value. If this instruction is used and further testing is carried out on a sample you will find that peaks have been shifted by the offset value. The lattice spacing values will also change.



This allows setting of the zero point on the readout scale to the true x-ray zero point, or adjustment of the scale to a peak position when using a particular calibration standard. The value given with this command should be the actual 2θ goniometer angle, in steps of 0.005°.

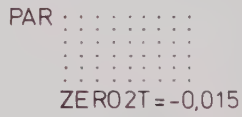
The zero point on the readout scale may not match the zero point displayed on the motor scale. The motor runs to its zero point on entry of the response to the prompt for selection of REMOTE or FRONT PANEL mode. The difference between the motor zero point and the readout zero point is given on the parameter, list as the value ZERO2T. This value can also be displayed on the readout by entering the command to display the 2θgoniometer angle, when the motor is at its zero point.



If the difference between the motor and readout angles is greater than 2.56° the following error message is displayed:

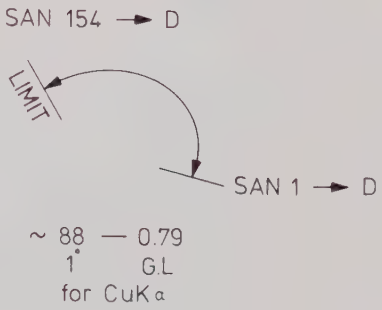
** 11 OUT OF RANGE

If this message is displayed it is necessary to recalibrate the system before continuing. Refer to the PW1710 Service Manual for recalibration information.

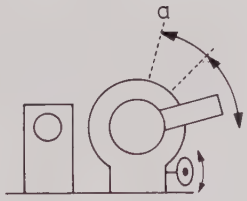


SAN a This instruction is used to set the goniometer angle to "a°". The 2θ value entered with this must be positive, and expressed in multiples of 0.005°. Failure to observe these rules causes output of the error message:

** 1 ILLEGAL ARGUM



The goniometer angular travel limit is quite high, with the actual value depending on the accessories fitted to the system. Normally the 2θ limit is around 155°. The system is fitted with an arm which closes a microswitch. This can be adjusted to set the limit required. Selection of a value above the microswitch limit causes the microswitch to trip, the goniometer stops and the following error message is displayed:

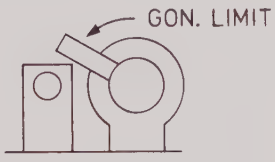


** 13 GONIOMETER LIMIT

On display of this message it is necessary to clear the error condition and then use the set the goniometer angleinstruction to reset the 2θ goniometer angle within the set limits. Remember that the lowest permissible level is 0°.

IAN a

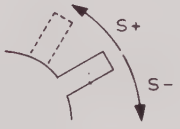
This command is used to alter the 2θ goniometer angle. The value "a°" is added to the current angle, thus a negative value decreases the angle. The change value, "a°", must always be a multiple of 0.005°. Be careful that the new angle required is within the limits set (see the set the goniometer angle command). When decreasing the 2θ value make sure not to specify a negative angle, as the following error message is output:



** 11 OUT OF RANGE

This instruction is normally used to:

- Go from peak to background locations on one or both sides.
- Make stepping routines in programs.

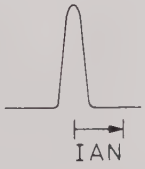


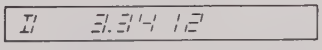
The system does have other facilities to perform these operations. Details are included in the following chapter.

DPL D

Display the value of the lattice spacing on the left. This value is calculated from the 2θ goniometer angle given on the readout. This calculation is based on Bragg's law:

$$2d \sin \theta = w$$





The expression "w", in the equation is the wavelength of the actual radiation line of the x-ray tube in use. This line is programmed using the set wavelength command. The current value for "w" can be found using the print parameters command.

The wavelength is expressed in Angstrom units, with the lattice spacing, "d", calculated and displayed for the current goniometer position. If the x-ray line wavelength is changed the value "w" must be changed accordingly. Refer to the set the wavelength command. The current wavelength can always be obtained using the print parameters command.

```
LWL 35 UPL 70
RFS 2E3
RTC .5 RSP 20
SPE 1.00 INT 0.05
STP 0.010 MXN 10
MXS 0.010 MXT 1.00
BGT 3.00 BGM 2
BGA 0.500 WAV 1.54186
SOF ROF DON POF
ZERO2T -0.150
```

The following examples demonstrate the effect that changing the goniometer angle has on the calculated lattice spacing value.

```
SAN 60          set 2θ goniometer angle to 60°
DPL D           display the calculated lattice spacing value
```

This example results in display of the lattice spacing value 1.54186

```
SAN 70
DPL D
```

This example results in display of the lattice spacing value 1.34408

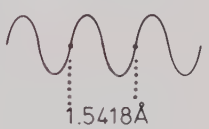
```
SAN 80
DLP D
```

This example results in display of the lattice spacing value 1.19935

WAV w This instruction is used to set the wavelength to the value "w". The current value of "w" can be printed using the print parameters command.

A new value must be set for "w" if a different x-ray K alpha line is used. For example, a new value must be set when another tube anode is used, or if you wish to study K alpha 1 or K alpha 2. For example "w" for Copper radiation is:

K alpha mean	1.54186
K alpha 1	1.54060
K alpha 2	1.54439



ANGLE ⟷ D

$2d\sin\theta=\lambda$ BRAGG

SAN xx
DPR G } =2θ 2T/2 ⟶ θ

WAV=λ
θ + λ known ⟶ d

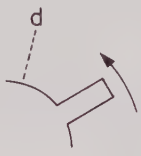
The following table gives values of "w", for other anode materials:

Anode Material	"w" (K alpha 1,2 in Angstroms)
Cr	2.29101
Mo	0.70173
Fe	1.93736
Co	1.79025

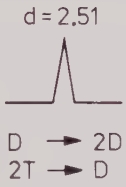
The wavelength value, "w", is also used when making continuous scans with peak labelling and when setting the goniometer angle to twice the arcsin of the wavelength divided by twice the lattice spacing. The set the goniometer angle, set the wavelength, and display the lattice spacing value commands also allow calculation of the lattice spacing values for peak positions after a diffractogram has been produced.

SAD d

This command is used to set the 2θ angle to a value calculated from the selected lattice spacing value. You should not select "d" values which give a 2θ value outside the goniometer travel limits. For example, for Cu K alpha, a value of "d" from 80 to 0.80 can be used.



The angle is set as close as possible to the calculated 2θ value, as a multiple of 0.005°. A value of "d" is calculated from this setting using Bragg's law, and displayed. This calculated value will be slightly different from the input, because of the 0.005° step.



The useful range must be determined by experiment. The recommended procedure is as follows:

- Give the correct value of the wavelength "w", using the set the wavelength command. Set the display using the display the lattice spacing command.
- Use the set the goniometer angle command to set 2θ to 1° and read the lattice spacing from the display.
- Use the set the goniometer angle command to set the maximum goniometer angle below the GONIOMETER LIMIT and read the lattice spacing again.
- Values outside this range, when found in literature on the subject material, may have been measured with a different wavelength, or calculated. Any lattice spacing value less than half the selected wavelength will cause display of the error message:

  ** 11 OUT OF RANGE

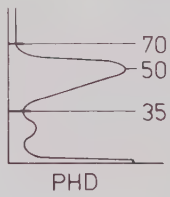
- The set the goniometer angle command is normally used to:
  - . Return to known peaks, or look for potential peaks.
  - . As a calculator converting the lattice spacing to 2θ effectively reversing the function of the set goniometer angle command.
- A first approximation for copper radiation can be calculated taking the 2θ lattice spacing as 90°.

2.3.3 DETECTOR

The function of the detector is to signal the intensities of the diffracted beams. It must be tuned to suit the x-rays being used. The main settings are done during installation. The upper and lower levels can be set separately.

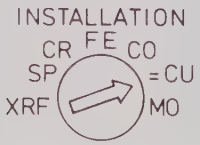
UPL 70
LWL 35

Set the upper and lower levels for the pulse height analyzer. The values in the examples are typical values for a PW1711 detector.

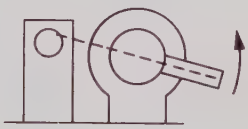


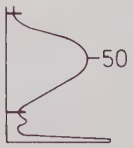
If the type of radiation (x-ray tube anode material) is changed refer to the section on the secondary control panel in the first chapter. If extremely high backgrounds occur on certain sample types refer to the previous section.

Always ensure that direct x-ray transmissions do not fall on the detector. This happens when 2θ is set close to zero. This is particularly important on equipment which does not close the shutter or have facilities to intercept the beam.



When using a PW1964/70 scintillation detector the lower and upper levels are farther away from the centre value. Check this by producing a pulse height distribution curve.

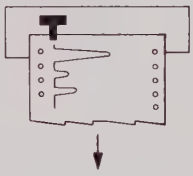




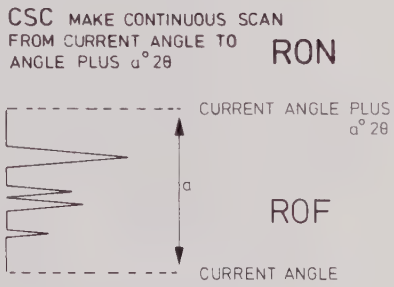
2.3.4 RECORDER

A recorder is normally used with the system to provide a permanent record of results, and to enable the interpretation of results which include fast variations in intensities which could not be followed on a display. The recorder enables production of a graph of these intensities. Normally the paper feed rate is linked to the rate of change of the diffractometer angle to provide a diffractogram.

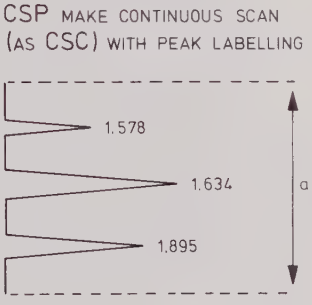
RON This instruction is used to switch the recorder ON. You must always use this instruction after switching the system ON, to enable production of graphs. It is not necessary to repeat it after the recorder is used unless the recorder off command has been used.



ROF This instruction is used to switch the recorder OFF. When this instruction is used the recorder is disabled and it is necessary to use the recorder on command before trying to produce any further graphs. In other words these instructions are used to enable and disable the other instructions which use the recorder.

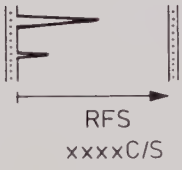


When using a PM8210 recorder the whole system must be switched OFF and ON again, after replacing the paper. This is necessary to reset the microprocessor controller in the recorder.



PON This instruction is similar to the recorder on command, and is used to enable the printhead on the recorder, if fitted.

POF This instruction is used to disable the printhead, on the recorder.



RFS iEj The recorder full scale command is used to set the recorder to record intensities across its full scale. When the recorder is operating it indicates the actual intensity like a meter, thus acting as a ratemeter. The record would not be very useful if travel only occurred across a limited segment of the scale. For this reason the full scale deflection can be adapted to the known (or expected) maximum intensity value. The range of possible settings is from 100 (1E2) to 500,000 (5E5) counts per second for full scale.

Full scale deflection values must be entered in exponential notation, as powers of ten. For example:

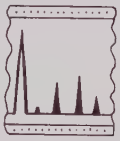
1E2 = 100

100,000 = 1E5

Twice or five times this range can be selected by substituting two or five for "i" as follows:

2E2 = 200

5E5 = 500,000

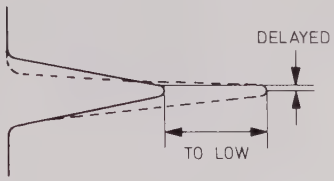


Intensities are detected across a wide range when generating a diffractogram. This instruction is very useful in that it can be used to study a specific region of interest, by setting the full scale deflection to suit, highlighting the required information.

RTC x This instruction is used to set the ratemeter time constant. The term ratemeter refers to the recorder which functions as a ratemeter when producing diffractograms.

All x-ray intensities have a basic fluctuation because of their physics. These fluctuations are called "counting statistics", and are shown on the ratemeter. It is difficult to read the intensity directly because of these fluctuations, both on the display where they change very rapidly, and on the recorder where the pen vibrates rapidly. Setting the ratemeter time constant allows acquisition of a clearer mean intensity by averaging the intensity over a specified time. The values allowed for "x" are 0.1, 0.2, 0.5, 1.0, 2.0 and 5.0.

RTC S This instruction automatically selects the best ratemeter time constant depending on the scan speed. This eliminates distortion of peaks, by setting a time constant that is small enough to prevent peak displays being lowered, and delays before displaying them. When the best constant has been calculated it is rounded to the nearest value from the available options. The equation used as the basis for this automatic constant selection is:



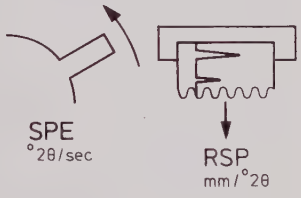
0.01 / Scan Speed (expressed as $2\theta^{o}$ / seconds)

RTC F This instruction selects the nearest constant option to:

10,000 / Recorder Full Scale Setting

This is coupled with the full scale and the time constant is selected according to the size of the counting statistics relative to the intensity. In addition the traverse speed of the recorder pen plays a role. This is 0.25 seconds for a full scale movement.

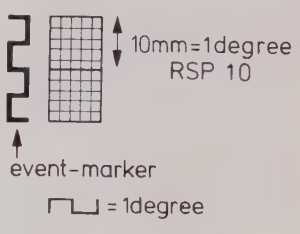
RSP This instruction is used to set the recorder paper speed. The speed of the paper is coupled to the diffractometer movement. The range of speeds is from one to 50 mm per degree. Well defined scales are normally used to allow the angular positions of peaks to be read, typically ten or 20 mm per 2θ degree.



SPE a This instruction is used to set the scan speed for a continuous scan. The scan speed is set in $2\theta^{o}$ / sec. This is normally used with the make continuous scan, the make continuous scan with peak labelling and the measure area from the current angle to the 2θangle a^{o} commands.

Select scan speeds that suit the information to be recorded. When the goniometer speed is changed the paper speed is automatically changed accordingly, ensuring that the paper feed speed remains constant relative to the angular speed of the goniometer. The value for "a" can be up to 1.27^{o} in steps of 0.001^{o} expressed with either two or three places of decimals.

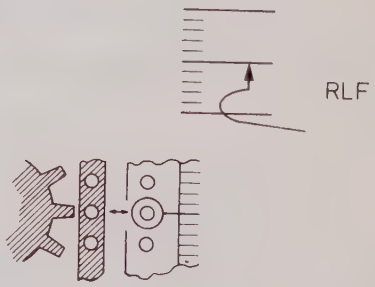
On the PM8203A recorder an automatic scalar marking is incorporated on diffractograms. This makes it easier to identify the 2θ values where peaks occur.



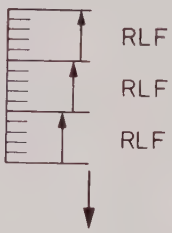
The product of the recording speed and the scan speed should not exceed ten mm per second when using the PM8203A or 16 mm per second when using the PM8210. The PM8203A speed selector should always be set to the ten mm per second maximum.

RLF

This command is used to advance the paper in recorders. On the PM8203A the paper is advanced until the next ten mm line is level with the pen. This command is particularly useful as most recordings are started with an integer value for 2θ. This makes it quite easy to identify $2\theta^{o}$ values, with characteristics on the diffractograms. Refer to the documentation supplied with the recorder if further information is required on any aspects of operations.



Multiple line feeds provide a useful method to separate diffractograms. Each line feed produces a five mm movement on recorders with a line feed facility. The PM8210 does not have the line feed facility, and in this case it is necessary to use a print command with a blank space for output to obtain a line feed.



RFS iEj The recorder full scale command is used to set the recorder to record intensities across its full scale. When the recorder is operating it indicates the actual intensity like a meter, thus acting as a ratemeter. The record would not be very useful if travel only occurred across a limited segment of the scale. For this reason the full scale deflection can be adapted to the known (or expected) maximum intensity value. The range of possible settings is from 100 (1E2) to 500,000 (5E5) counts per second for full scale.

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3.6 STEP SCANNING 3-13

3.1 INTRODUCTION

Scanning over an angular range can be carried out continuously or in a series of steps. This chapter details the instructions needed to control scanning. Once again example programs are included with the text ensuring familiarity with the form programs take. This will help when going through the chapter on programming.

3.2 CONTINUOUS SCANNING TO PRODUCE A DIFFRACTOGRAM

This section introduces the instructions needed to carry out a continuous scan and record the results. Each of the commands is explained and examples of use are given. Always follow the instructions given in the previous chapters before attempting to use these commands.

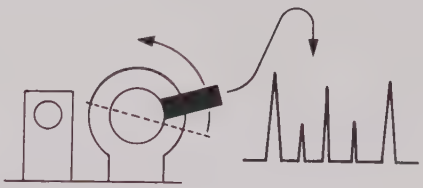
SAN a This instruction is used to specify the 2θ goniometer angle where the scan is to start. The value "a" refers to the required angle, and must be expressed as a multiple of 0.005^{o}. Scans should start with an integer value for 2θ, to make it easier to interpret the results.

SPE a This instruction is used to specify the scan speed. The 2θ goniometer speed per second is given by the value "a". The speed range available is from 0.001^{o} / sec to 1.27^{o} / sec. Speeds from 0.13^{o} / sec must only have two characters after the decimal point. Entry of more than two numbers after the period, in this higher range, result in display of the following error message.

** 1 ILLEGAL ARGUMENT

Speeds must be choosen to suit the purpose of the scan. The quality of diffractograms is lower at high speeds because of the rapid changes in the detected values.

When a high scan speed is selected, the product of SPE and RSP should not be greater than the maximum speed of the recorder (10 mm per sec for the PM8203A; 16 mm per sec for the PM8210).

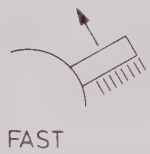


A scan at one degree per second allows a preliminary look at a diffractogram, giving an impression of how many peaks there are, their distribution and their heights.

INT t This instruction is used to set the sampling interval times. The value entered for "t" must be a multiple of 0.005 seconds. When making a continuous scan with the make continuous scan command the sampling interval time must be set to zero.

The minimum time constant is 0.1 seconds. When the set recording time constant command is given with an "S", and the recorder speed has been set to one mm per second, the calculated time constant of 0.02 seconds will not be used. The recorder takes 0.25 seconds to cover the full scale, resulting in peak distortion at high speeds. Counting statistical variations in the background region are also shown. Less smoothing occurs in the background.

CSC a This instruction is used to start a continuous scan, from the current 2θ goniometer angle to an angle "a^{o}" higher. Take care to ensure that the end angle specified by entry of this instruction is within the goniometer travel limits. The range can be from 0.005^{o} to 163^{o}.



FAST

SLOW

FAST

CSP a This instruction is used to start a continuous scan, from the current 2θ goniometer angle to an angle "a^{o}" higher, with labelling of detected peaks. Take care to ensure that the end angle specified by entry of this instruction is within the goniometer travel limits.

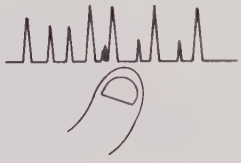
The following example demonstrates the way in which the commands explained so far can be used. The lower case text is explanatory, and is not displayed.

WAV 1.5418	correct the wavelength
LWL 35	set the lower level
UPL 70	set the upper level
RON	enable recorder output
RFS 2E4	recorder full scale 20,000 counts / sec
RTC S	set recording time constant using the formula detailed previously
RSP 10	set recorder paper speed to 10 mm per 2θ degree
RLF	recorder line feed
DPL X	display the intensity on the left
DPR G	display the 2θ goniometer angle on the right
POF	turn the printer OFF
DOF	stop dual output
SON	turn the spinner ON (if available)
SAN 15	set the 2θ start angle to 15^{o}

The facilities detailed and demonstrated above are useful for the following applications:

- They allow selection of the deflection scale to be used on the recorder. This allows selection of an appropriate scale, depending on the peaks to be viewed. Remember that the time constants lower the peaks and in slower scans they will appear 1.5 times higher.
- Compact diffractograms recorded at high paper speeds can be very useful providing "finger-print" identification. For example with the paper speed set to 1 mm per $2\theta^{o}$ the output resembles the film from a large Debye Scherrer camera.

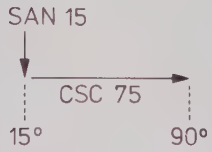
If greater detail is required it is necessary to set the recorder paper speed to a lower rate. Normally the speed is set to equate 10 mm or 20 mm with each degree.



3.3 CONTINUOUS SCANNING TO PRODUCE DIGITAL OUTPUT

Digital output can be obtained from a continuous scan and monitored on any of the following:

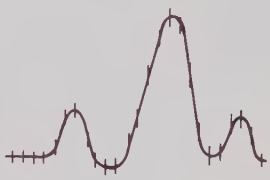
- The operator's terminal.
- The front panel readout.
- The dual output terminal.



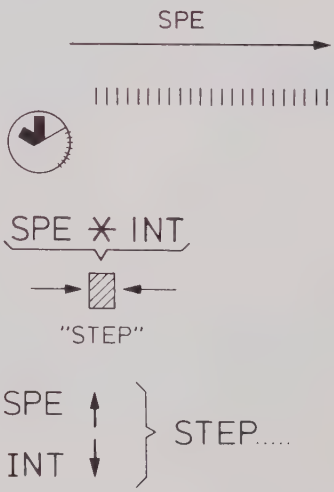
Digital output is obtained by setting the sampling interval time to a non-zero value. The preparatory steps are as detailed previously.

INT non zero This instruction is used to set the sampling interval time in seconds. The counts are sampled during scanning. When the interval time has elapsed, the current angle and the counts are printed. Counting then starts again for the next interval. Thus the goniometer moves during counting.

The angular step over which counts are collected is the product of the scan speed and the sampling interval time. When high scan speeds are selected with short sampling interval times a large amount of output is obtained. The goniometer and recorder stop from time to time, allowing the data transfer to terminals to catch up.



Values for the sampling interval time must be multiples of 0.05, in the range 0.05 to 3276. They must be set after the scan speed. It is very important to change the interval time after changing the scan speed in order to maintain the same step size.

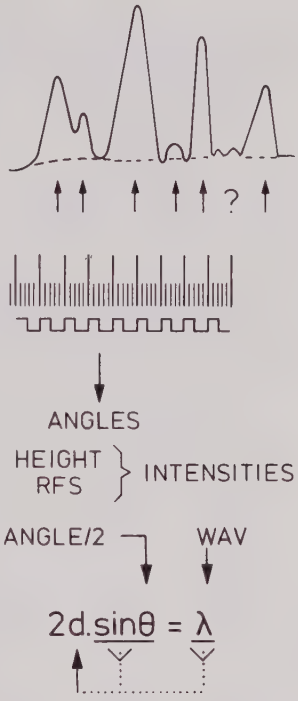


3.4 CONTINUOUS SCANNING WITH PEAK LABELLING

When a continuous scan has been made using the make continuous scan command the output on the recorder is a diffractogram. It is possible to perform a continuous scan without producing a diagram, by disabling the recorder. Failure to get any results may be because the sampling interval time has been set to zero, the x-ray shutter is closed, or the scan started on a peak. The continuous scan with peak labelling allows determination of peak angles, their d-values and raw intensities. The diffractogram must be interpreted, to identify the background and the peaks. Interpretation of the diffractogram should be as follows:

- Determine the peak position by reference to the grid divisions and the scale, keeping the starting angle aand recorder paper speed. Measure the height of peaks, keeping possible distortion in mind, especially the height on fast scans.

- Convert the angles read off the chart into lattice spacing values using tables or calculators. Bragg's law is normally used to perform this conversion. Scale the intensities relative to the highest. This must be done in order to format the data to simplify the identification search through the J.C.P.D.S. file.



The continuous scan with peak labelling routine tracks the intensity during recording. Measuring points are temporarily stored, continuously being replaced by the following ones. The results are not stored, the only product being the output. Thus long range operations like background subtraction and scaling of the intensities are not available. The following example demonstrates a typical series of preparatory steps before carrying out a continuous scan with peak labelling:

WAV 1.5418	set correct wavelength
LWL 35	set lower detector level
UPL 70	set upper detector level
RON	recorder ON
RFS 2E4	set recorder full scale to 20,000 counts / sec
RTC S	set recording time constant using the formula detailed previously
RSP 10	set recorder paper speed to 10 mm per 2θ degree
RLF	recorder line feed
DPL X	display the intensity on the left
DPR G	display the 2θ goniometer angle on the right
PON	turn the printer ON
DON	start dual output
SON	turn the spinner ON (if available)
SAN 15	set the 2θ start angle to 15^{o}

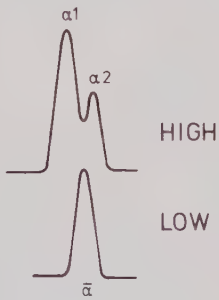
The following text provides a more detailed explanation of the instructions used in the previous example.

WAV w The wavelength given in the example is the value for the weighted mean of K alpha 1 and K alpha 2 of copper radiation. This value is used to convert angles to lattice spacings. This is the best one for the low angle region.

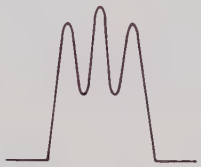
In the higher angle region the alpha 2 peak moves away from the alpha 1. In this case it can be located using the make continuous scan with peak labelling command. Two lattice spacing values are obtained instead of one.

Detection of two peaks occurs because, as previously mentioned, measurements overwrite previous values. If two peaks are detected, with the second at half height they are taken as alpha 1 and alpha 2. If, however, the second is higher, or there are three, the first is alpha 1 and the second is alpha 2 + alpha 1 of the following reflection, and further interpretation will occur.

Higher region measurements can be repeated with K alpha 1 (1.54050) or with K alpha 2 (1.54434) in important cases. Alternatively scans can be divided into two sections, using the set the wavelength command in between sections.



SAN 15 The start angle should always be in a background region. If a scan starts at a peak, stop it; change the start angle and begin again.



SPE 0.1 This instruction sets the scan speed in 2θ degrees per second.

INT 0.5 This instruction sets the sampling interval time in seconds.

The scan speed and sampling interval time are also used when carrying out ordinary continuous scans, but they are more important when labelling peaks. The combination of these values is also critical.

If a peak scan is started, over 75°, the diffractometer will start moving at a speed of 0.1° per second (6 degrees per minute). The signal from the detector will be recorded in the normal way. The counts will be sampled at intervals of 0.5 seconds. After running over a 0.05° step a number of counts are taken. The step size is determined by the product of the scan speed and the sampling interval time. This has previously been described for digital output from continuous scanning.

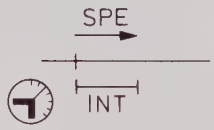
Continuous scanning with peak labelling uses these digital values to determine when peaks are found. It does not look at the recorded signal. Peaks can be defined in the following manner:

- The highest point.
- A number of ascending steps followed by a number of descending steps.
- An increase followed by a decrease.

All these methods introduce smoothing at varying levels depending on the parameter settings. A further definition can also be derived:

- The point where the slope of the line changes from positive to negative.

The continuous scanning with peak labelling routine calculates the second derivative of the curve slope to identify peaks.

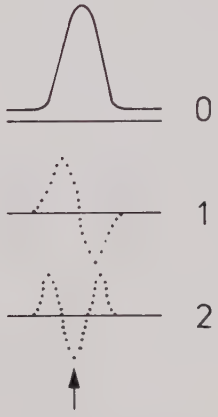


"STEP" = SPE ✳ INT

When measuring intensities there is a spread in the values obtained, centering around the mean value. The standard deviation provides a measure of the spread, with its value given by the square root of the number of counts of that mean. Two thirds of the results will fall within one standard deviation of the mean, in other words in a band twice one standard deviation width. A band four standard deviations wide will enclose 95% of the measurements, although the spread can be over six standard deviations.

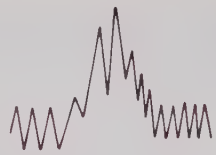
When carrying out a continuous scan with digital output these variations can be seen on the output and on the readout. It is not valid to define a peak as a point higher than its immediate neighbours for this reason. If peaks were identified by lower neighbours a large proportion of the steps would be identified as such. Further description of peaks can be:

- Peaks should consist of a group of higher values.
- The points should occur close to one another giving narrow peaks.
- The high points should be much higher than the statistics.

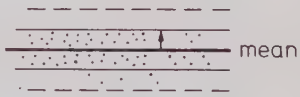


The continuous scan with peak labelling routine uses the first nine steps to look at the statistics. A threshold is calculated from these nine counting results. Later variations are then compared with this threshold.

The background level is not calculated. The object is to identify real variations in the background regions. Any variation greater than this level is marked as a peak. The sensitivity of the continuous scan with peak marking routine is optimised for the background regions in this manner.

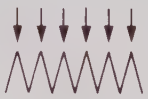


It is important that the first nine steps are only in the background. With a step size of 0.05 they cover 0.4°.

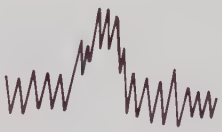


When the threshold has been defined the routine starts looking for peaks. The second derivative is used to monitor the data trends. The last nine readings are always used. They shift along as the diffractometer moves. Smoothing is included to damp counting statistics.

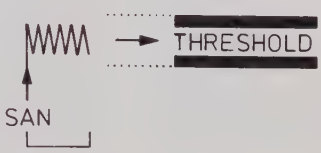
In the example each group of nine steps covers an angular range of 0.45°. The routine is tuned to peaks of this width, as measured at half height. This applies to a lot of diffractograms.



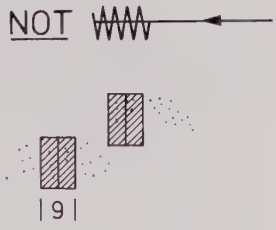
Wider peaks will not be seen as the variation is then too slow. Where wider peaks occur steps of 0.1° or 0.2° may be more suitable. Alternatively narrow details, as encountered at overlapping peaks, may have been smoothed away. The step can be reduced to 0.03° to make more detailed studies of narrow peaks.



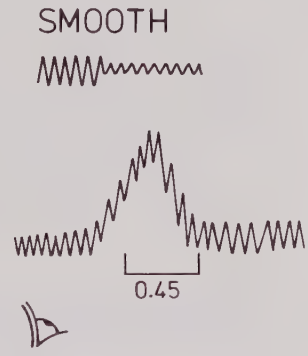
It is not advisable to use steps smaller than 0.03° because shoulders on diffractograms may be interpreted as peaks. This allows detection of an alpha 2 coming out at the side, but statistical variations are large at the peak level, as can be demonstrated by taking the square root of a large number. The sensitivity is tuned to the background situation. Counting statistics affect the routine when a narrow step is used and non-real peaks are output at the sides.



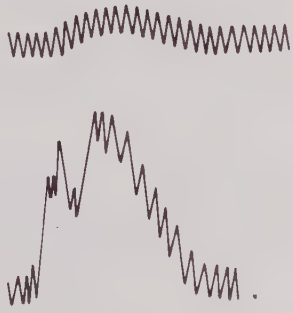
On detection of a peak the routine calculates the top location. This is done by fitting a parabola on five points around the top. The result, as printed, shows more detail than the 0.005° goniometer step. The routine incorporates a facility to make corrections if counts have been sampled during goniometer movement. Values belong to the centre of intervals.



The lattice spacing is calculated from the angle of the centre of the interval, using the current wavelength value. The intensity is also determined using a parabolic fit. Ensure that there is no background subtraction. The parabolic fit procedure has been selected to give a closer approximation to a single top. Do not expect it to work perfectly on other shapes such as shoulders however.

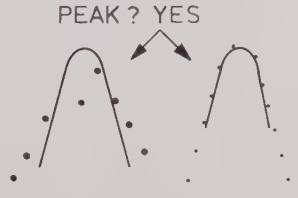


When a scan is repeated the values tend to vary slightly, both the sensitivity (threshold) and all the following steps.Interpretation of marginal peaks also varies slightly on repetition of scans.



The results of a continuous scan with peak labelling are normally visibly different from a diffractogram. This is because smoothing is effected in different ways and no threshold is used on continuous scans with peak labelling. It is also important to remember that a nine step window is used for labelling meaning that single step peaks are ignored.

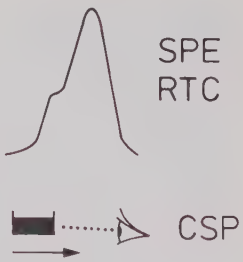
Do not look for the differences after producing a diffractogram and using the peak labelling facility. Combine the duplicated information obtained and use the more clearly defined angular values.



A lot of combinations of the scan speed and sampling interval time can still be used, even when a step size of 0.05° is selected, as shown in the following examples:

(1.2°/m)	SPE 0.02	... INT 2.5
(2.4°/m)	SPE 0.04	... INT 1.25
(6°/m)	SPE 0.1	... INT 0.5
(30°/m)	SPE 1	... INT 0.05

When using a low sampling interval time, such as 0.05 seconds, 20 samples are taken each second. The number of counts collected will only be $1/20^{th}$ of the intensity, in counts per second as shown on the recorder. In other words with a background of 400 counts per second 20 counts are taken per reading. With the counting statistics this may vary between ten and 30.



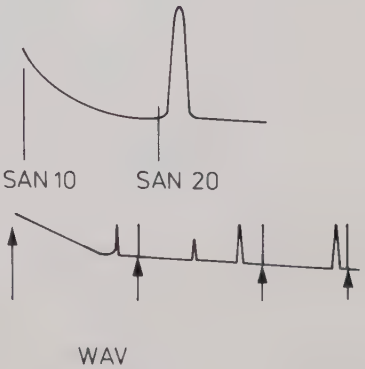
The sensitivity threshold, as derived from the first nine steps, will also be less clearly defined. In combination with the less accurate steps, accuracy in the background region may be poorer. Certain safety factors have been included for this reason.

When using a lower speed and a larger sampling interval the number of counts per reading is higher. In the absolute sense the standard deviation value also increases, but the relative variation is less because the spread forms a narrower band around a better mean. In this sense results become more reproducible.

When a slope is present in the background a high start results in a higher threshold, thus lowering the sensitivity. If no peaks are found in the high region, start again at a lower position. Alternatively couple a number of continuous scans with peak labelling. This can be done within a program (see the chapter on programming). When using such programs ensure that all starts are in low regions.

When starting again, at a higher angle, the wavelength can be set to the K alpha 1 value. This is also programmable. Ensure that such operations are clearly marked on the output.

When a number of continuous scans are carried out sequentially, either within a program or using the repeat program command, as detailed later, the type of recorder is significant. A continuous diffractogram can be produced on the PM8203A, as long as no line feeds have been given. Certain other recorders introduce additional chart movements for label printing.



3.5 OUTPUT FROM CONTINUOUS SCANNING WITH PEAK LABELLING

The following devices can be used for output of continuous scans with peak labelling:

- Operator's terminal and second output device (DON).
- Printing recorder PM8210.
- Front panel display.
- Front panel control.

If the first of the above options is selected the output displayed on the operator's terminal and the other output device is a list under the following headings:

ANGLE D-VALUE INTENSITY

The angle is given to three decimal places, as calculated by parabolic fitting. This differs from results obtained using the search for maximum intensity and the search for the top intensity and subtract background modules.

Lattice spacings are given to four decimal places. This does not mean that accuracy to four decimal places is always obtained, especially in the lower angle range.

The intensity is given in the third column in counts per second. They are extrapolated from the parabolic fitting although the original readings are taken over the specified sampling interval time. The intensity values are raw intensities without any background subtraction.

If the system includes a PM8210 recorder, it can be used as a second output device by using the enable printhead command. The recorder can then be used in a similar manner to the second output device, for output of text, headings, and program listings. It can also be used to label either continuous or step scan outputs. It is important to remember that the lines appear in reverse order because of the direction of the recorder paper transport.

When the continuous scan with peak labelling routine is used the dual output and printhead enable commands have different effects. On detection of a peak the dual output devices list the angles, d-values and intensities. The d-value is stored in a special buffer, and when the recorder pen has moved over ten millimetres, it is printed next to the peak.

This means that the bottom of the figures is at the peak, as near as possible, within the tolerances of the recorder. There are nine possible positions, equally spaced across the chart (tabs). The nearest tab is selected, depending on the intensity, and relative to the full-scale setting. When labels have to be printed, the scan is stopped to enable proper positioning of the label, especially with tabs towards the right of the recorder.

When using a high speed with a high recorder time constant, the peak may appear lower than the real intensity. Since the tabs are discrete positions, some space may result. Spaces of this type can also occur for other reasons. Sometimes peaks occur close together, for example alpha 1 and alpha 2, or overlap situations.

A recorder speed that condenses the diffractogram can also result in detection of a second peak before the label on the first peak is printed, because of the ten millimeters gap between the printhead and the recorder pen. A buffer which can hold up to four peak values is used, therefore if more than four values are detected they will be printed on the output terminals but not on the PM8210 printing recorder.

The second peak may be the same height. If this happens the second label jumps one tab sideways. In this event do not look at the tab location, but at the bottom of the figures to associate printed labels with their peaks. At full scale the tab jump is downwards in order to avoid overprinting. The tab will also jump up and down, if allowed, when four close peaks are detected in the lower region.

On completion of scanning there may still be some labels to be printed. The chart will advance the required distance and stop when all the labels have been printed. For this reason it is not advisable to perform a series of scans with peak labelling, without including a number of line feeds to indicate the gaps.

When front panel display is selected to run a continuous scan with peak labelling, with the display coupled to the output buffer, the successive output lines are shown on the display. Each new line replaces the previous one and scanning is continuous.

When front panel control is selected a special form of the continuous scan with peak labelling routine is performed if the following conditions apply:

- The system is fited with a PM8203 and the printhead has been disabled.
- Dual output has been disabled.
- The readout display has been set to output the buffer contents on the right hand side and leave the left side blank.

When the first peak is detected the readout shows the angle, the lattice spacing value and the intensity expressed in counts per second. The scan then halts permitting recording of the displayed values. Pressing the ENTER pushbutton on the front panel starts scanning again, and it continues until detection of the next peak, then the same procedure must be repeated. This type of "interrupted" continuous scan can be used whether or not other peripherals are fitted, as long as they are disabled when present.

3.6 STEP SCANNING

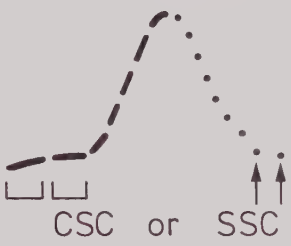
The system incorporates facilities to perform a step scan. This enables stepping through a part of a diffractogram, for instance over a peak, in order to get a detailed picture of its shape. The output is normally required in digital form to allow further processing of the information obtained. This processing of information is normally performed by a computer. It is also possible to produce a complete diffractogram, in steps, although it can be very time consuming.

Basically, step scans and continuous scans can handle the same diffractogram. In the step scan, however, counts are only collected and output at exact angular positions. On the other hand continuous scans follow and record the intensity. This means that undistorted images can only be obtained at low speeds. The image produced by a continuous scan is smoothed and not digital.

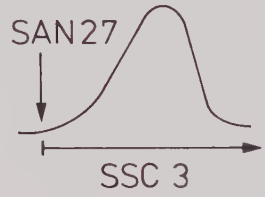
On selection of digital output by entry of a non-zero sampling interval, counts are sampled during movement, and not at discrete angular positions. This allows generation of a mean value over an interval in a relatively short time.

SSC a

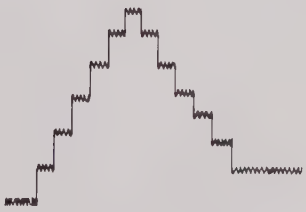
This instruction is used to start a step scan, from the current 2θ goniometer angle to an angle "a°" higher. Take care to ensure that the end angle specified by entry of this instruction is within the goniometer travel limits. The range can be from 0.005° to 163°.



When the sampling interval is non-zero a stationary measurement is carried out at the starting angle, the goniometer traverses the specified step and the next measurement is made. The cycle continues in this manner until the specified angular range has been covered.



The system outputs each angle and the measured counts. These counts are the values sampled when digital output is specified, and are not converted to intensity. Intensity in counts per second is only obtained when the sampling interval time is set to one.



The following example demonstrates a typical step scan. It includes the setting of all the necessary parameters. The recorder must be enabled. The paper transport is calculated as the product of the scan speed and the recorder paper speed. The recorder moves continuously, so there is no coupling to the goniometer or the output waiting time. The plot is stepped with an increment that is slightly larger than the product of the scan speed, the recorder paper speed and the sampling interval time. It is also necessary to set the recorder full scale value and the ratemeter time constant must be set using the automatic constant value calculation detailed previously. The following example demonstrates a typical use of the step scan facility.

RON	recorder on
SAN 26	2θ goniometer angle 26° (for quartz sample)
SPE .5	set 2θ° / sec scan speed .5°
RSP 2	set recorder paper speed to 2 mm / 2θ°
RTC S	set ratemeter time constant S
RFS 5E4	set ratemeter full scale to 500,000 counts per second
STP 0.03	set the 2θ step size 0.03°
INT 2	set the sampling interval time to 2 seconds
SSC 1	perform a step scan from 26° to 27° 2θ

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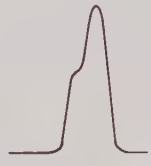
4.1 INTRODUCTION

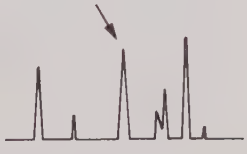
When a complete diffractogram has been produced it may be necessary to study a single peak in more detail. This may be necessary for any of the following reasons:

- To ensure the presence of a peak.
- To get a more precise definition of the peak location.
- To study the peak profile.
- To use the intensity for quantitative analysis.

There are a number of facilities available, including scanning techniques. Peak identification and location can be effected using the continuous scan and the continuous scan with peak labelling routines. Individual profiles can be studied using the step scan facility. These facilities are detailed in the previous chapters.

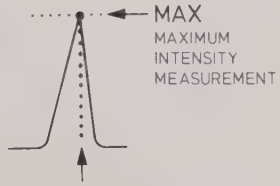


CSC,CSP
SSC



This chapter details the search for maximum intensity routine, the search top intensity around current angle and subtract background routine and the measure area from current angle to the 2θ angle "a°". It is possible to write programs to perform these functions using the programming instructions. Further information on how to write programs is provided in the chapter on programming.

The measure area and search for maximum intensity routines are for routine quantitative analysis. They do not give direct output, as some data handling and output formatting is normally required.



4.2 SEARCH FOR THE MAXIMUM INTENSITY

The following parameters must be set before using the search for maximum intensity routine. They are detailed in the following text.

SAN a — This instruction must be used to specify the start angle "a°". This defines the region to be searched for maximum intensity.

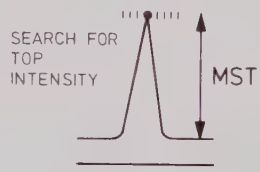
MXN n — This instruction specifies that "n" measuring points are to be used on each side of the specified start angle. This means that a total of twice "n" plus one points will be measured.

MXS a — This instruction specifies the size of the step between the measuring points, as "a°".

MXT t — This instruction specifies the measuring time at each point, as "t" seconds.

MAX — This instruction starts execution of the search for maximum intensity. A step scan is made around the specified start angle. The last three readings are added together and the average calculated after each step. The system looks for the highest value of this moving average.

The central angle and the intensity are calculated using the highest detected value. The data is only sent to the operator's terminal, and the goniometer is set at the calculated angle. The display left and right commands can then be used to view the results on the readout.



When the last value is the highest, an asterisk, "*", is added. When operating in FRONT PANEL mode it is advisable to do a second search for maximum intensity as the asterisk is not displayed. When the first result is at the end of the specified range, the peak will be seen to have moved when the second search is performed.

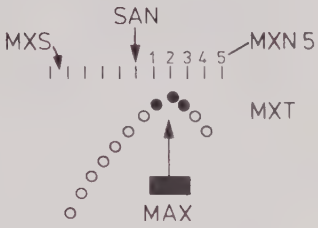
When uncertain about the probable peak position increase the number of points or the step size in order to search over a greater range. With clearly defined peaks the time at each point can be reduced to 0.1 or 0.05 seconds. The accepted values are as shown in the following example:

MXN 2 to 100 measuring points on either side

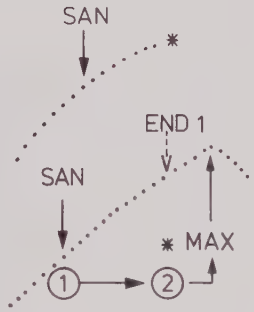
MXS 0.005 to 2θ degrees between the measuring points

MXT 0.05 to seconds at each point

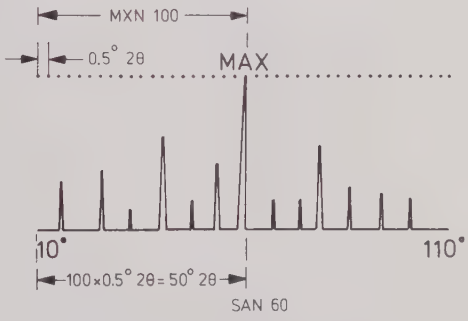
These ranges permit searches across a full diffractogram, by selection of 100 measuring points on each side of the start angle and setting the step size to 0.1^{o} or 0.05^{o}. With a 60^{o} start angle, 0.05^{o} step size and 100 measurements the search runs from 10^{o} to 110^{o}.



If the above procedure is adopted it is normal to reset the step size and number of points, to concentrate on the area of interest. It is faster, however, to use the continuous scan with peak labelling facility if a large angular range must be covered.

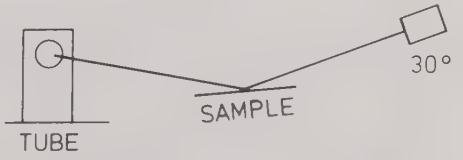


The search for maximum intensity routine is useful because detected peak locations can be offset for a variety of reasons. The height of the sample is critical, and the surface must be located precisely on the diffractometer axis. Samples are clamped from below, against a reference surface, because of this.

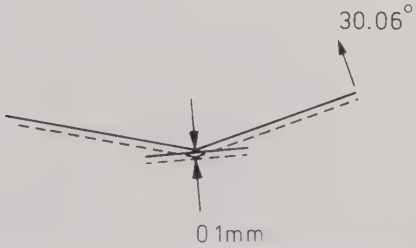


MXN 100 : 100 MEASURING POINTS ON EACH SIDE
MXS 0.5 : STEPS OF 0.5° 2θ

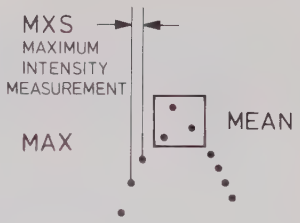
If the sample is 0.1 mm above the holder, peaks will be displaced. The displacement is about $+0.06^{o}$, using a 2θ goniometer angle of 30^{o}. If it is 0.1 mm too low the displacement is about -0.06^{o}. Therefore, when measuring a series of samples, variations of 0.1^{o} can easily occur.



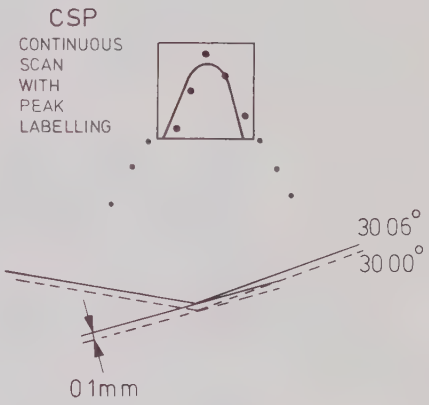
The main difference between the search for maximum intensity and continuous scanning with peak labelling routines, is that searching uses the results of measurements at discrete positions, and averages over three results to give a smoothing effect. The resultant highest point is in one of the step positions.



The two routines use different methods to perform approximations. Continuous scanning with peak labelling performs calculations while running, and gets a value over a step of a selected size, 0.05° for example. It then performs a parabolic fit over five results.



All measurements are also influenced by the counting statistics. It is also possible to adapt the diffractometer angle readout to a particular class of samples, with a given error, by combining the correct readout angle command with the search for maximum intensity routine.



4.3 TOP INTENSITY MEASUREMENT

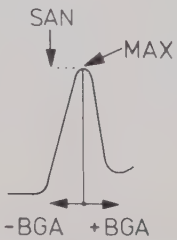
The top intensity measurement routine is derived from the search for maximum intensity routine. The same parameters used with the search facility must therefore be set, when using this routine. It is also possible to measure net intensity, subtracting the background when necessary. This means that further parameters must be set. This is demonstrated in the following example.

SAN xx.xxx	anticipated angle of top
MXN 10	10 measuring points at either side
MXS 0.01	step size between the points
MXT 1	measuring time at each point
BGM 2	enter background mode
BGA 0.5	set the background offset in degrees
BGT 3	set the background measuring time
MST	start execution of the top intensity routine
PTR 0	print result (net intensity)

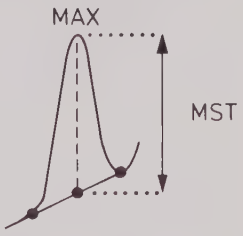
When this routine is used it measures the top range in exactly the same manner as the search for maximum intensity routine. It looks for the highest point, using a moving average of three points. When the highest point has been found the routine looks at the background mode. The background mode commands are explained in the following paragraphs.

BGM 2 When background mode "2" is selected two background measurements are made on each side of the peak.

BGA The background position can be selected using the set background offset in degrees commmand. The background offset, in degrees 2θ, is relative to the maximum found, and not the selected starting angle. This compensates for slight background shifts, owing to changes in the specimen surface height.



BGT t This instruction is used to set the background measuring time for each of the background measurement points.

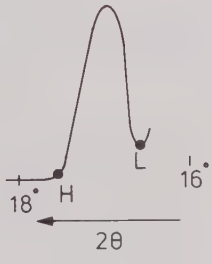


The mean background value is subtracted from the intensity of the top. The result is stored in the accumulator and must be printed and/or saved into a register before doing any other measurements. All calculations are performed on intensities, in counts per second. The measuring time and the background measuring time can be set to desired values, but it is important to consider the relative precision obtained when counting statistics are involved.

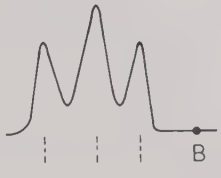
BGM L When the background is less clear on BGM Hone side of the peak it is possible to use only one measurement. This can be on either the low side, "L", or the high side, "H".

BGM 0 When the background mode is set to "0" (zero) the background is not measured. This can be useful in the following situations:

- When the background can be neglected relative to the peak intensity, thus saving measuring time.
- When the relative intensity of the background and the peak have been covered properly by an intercept in a concentration formula.
- When it is necessary to store the raw intensity to perform calculations on standard deviations.
- When the angular value of the top, or its lattice spacing, are to be stored for specific applications.
- When it is necessary to combine a single user-made background with several peaks.
- When the background is also required for statistical calculations.



The measure top intensity routine is blind, only displaying a result in response to an output command. Normally there are further data handling procedures, for example conversion into concentration, before the output is displayed. However the top may have been located at one end of the search range. In this event it is likely that the real top has been missed. If this occurs the procedure will continue with the background and calculate its result. Be careful when selecting the number of points to be measured and the step size.



When the peak intensity measurement routine is used in background mode "0" the routine returns to the top intensity giving the following:

- The intensity, in counts per second, in the accumulator.
- The intensity, in counts per second, in the intensity register.
- The highest sum of three steps, in the counts register.
- The angular position.

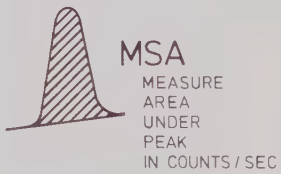
When the peak measurement routine is used, and the background mode is not "0", only the last background data is available. The top intensity measurement facility can be used for quantitative determinations. It is also possible to use the area measurement routine.

With five measurements taken each side, a step size of 0.02^{o}, and a sampling time of two seconds, a total of 22 seconds is required. The effective measuring time, the time spent on measurements that are used, is six seconds.

With ten measurements taken each side, a step size of 0.01^{o}, and a sampling time of one second, a total of 21 seconds is required. The effective measuring time, the time spent on measurements that are used, is three seconds.

4.4 AREA MEASUREMENT

This routine provides facilities to measure the (net) intensity of a peak. The total number of counts under a peak profile is collected in a scan. If required the background can be measured and subtracted. The resulting intensity is given in counts per second, stored in the accumulator, available for printing, or storage. The total time is given by dividing the scan range by the speed and adding the background measuring time. The following points should be taken into consideration when using this facility:



- The result looks small because of the PW1710 convention of converting the result to a counts per second value. As a scan takes a few seconds the mean value obtained with this routine is small, compared to the top value obtained with the search top intensity routine. This routine which is detailed later on is, however, no less accurate. It may be necessary to specify that decimals are to be shown, but this has no influence on the accuracy of the calculation, just the display.

- Greater accuracy can be obtained with a lower scan speed. More counts are collected, decreasing the relative error. The measuring time increases in the same manner as the counts, thus producing the same value as previously. When the result is converted into concentration, by multiplying with a (calibrated) factor, recalibration is unnecessary because of scan speed changes.

- When the result is used in a calibrated conversion to concentration, as detailed in the previous paragraph, it is necessary to recalibrate if the scan width is changed. Normally the results of this type of routine are expressed as total counts. On the PW1710 the result is expressed in counts per second. This means that the value obtained over a period greater than one second will be smaller, for a given sample. Thus it is necessary, if changing the scan length, to include a further calculation multiplying the result by the range. The product of the result and the scan range is of course insensitive to the length and speed of the scan, as allowances are incorporated in the routine.

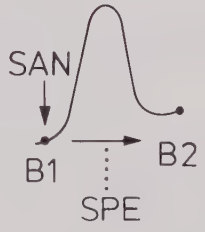
The area measurement routine uses some of the same parameters used with the search routine. These parameters must therefore be set before using this routine. This is demonstrated in the following example:

RON/ROF	depending whether recording is required, and if it is the recorder full scale setting, time constant, speed and line feeds must be set
SAN xx.xxx	set start angle to lower peak area angle
SPE .1	set count collection scan speed (in degrees per second)
BGM 2	select background mode "2"
BGT 3	set background measuring time of three seconds.
MSA 0.9	collect net intensity over 0.9^{o}
DEC 0	no decimals in output
PTR 0	print result
STR n	store result in register "n" for further use

BGM When the background mode is set to "2", the background is measured on both sides. The procedure starts with a background measurement which lasts as long as specified by the set the background measuring time command. The scan is then made, followed by the second background measurement. The mean value of the two backgrounds is then subtracted.

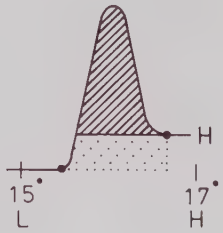
All calculations are done on an intensity scale, and the resulting net intensity can be taken as the area above the connecting line between the two backgrounds.

BGM L
BGM H When the background mode is set to low, "L", or high, "H", only one background level is measured. This value is then subtracted. The net intensity is the area above a horizontal line through this background point.



BGM 0 No background subtraction occurs when the background mode is "0". This mode is useful in the following circumstances:

- When the background can be neglected relative to the peak intensity, thus saving measuring time.
- When the relative intensity of the background and the peak have been covered properly by an intercept in a concentration formula.
- When it is necessary to store raw data to be used to perform statistical calculations.
- When it is necessary to combine user-made backgrounds with area measurement. This is sometimes necessary when scans must start and finish at points which which do not descend all the way to the background level.
- When the background is required for use with several peaks.



On completion of this routine the following data is available:

- The net intensity result, in the accumulator, in counts per second.
- The last measurement data in the counts and intensity registers. If the background mode is "H" or "2" this is the second background. If the mode is "L" or "0" they hold the integral scan.

The following commands are often used to get access to the values detailed in the previous paragraph.

RIN This command is used to recall the intensity to the accumulator.

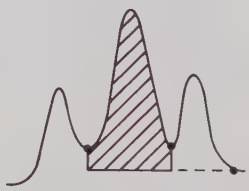
RCT This command is used to recall the counts to the accumulator.

PTR 0 This command is used to print the contents of the accumulator.

Both the measure area and the search for top intensity routines are provided to get a net intensity for a peak, for quantitative analysis. In such operations the precision is important, but the total analysis time may also be important. It is also important to get a lot of counts, as the relative error due to counting statistics decreases with an increasing number of counts. This is summarised in the following two paragraphs.

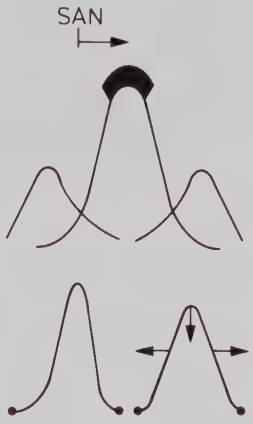
MST This is used to start the search for top intensity:

- In order to take a large number of counts.
- To search for the top, counteracting peak shift effects.
- To avoid possible overlap in the tails below peaks.
- To carry out a search for top intensity which is sensitive to the profile of the peak.



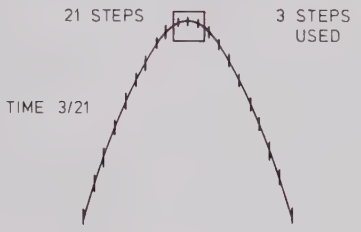
MSA This is used to start the area measurement, and has the following features:

- It is not sensitive to shape changes.
- It has a low sensitivity to shifts because the ends are at the background level, possibly touching overlapping neighbours.
- It gets less counts in the tails, irrespective of time spent there.

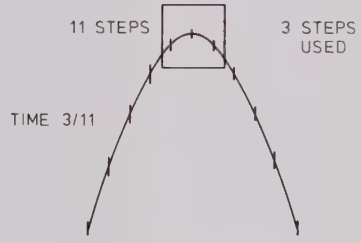


When making routine measurements using the top intensity measurement routine it is important to consider the counting statistics. The search for top intensity uses three values to calculate the top value. The following examples demonstrate this.

- With five measurements taken each side, a step size of 0.02^{o} and a two second sampling time, a total of 22 seconds is required. The effective measuring time, the time spent on measurements that are used, is six seconds.



- With ten measurements taken each side, a 0.01^{o} step size, and a sampling time of one second, a total measuring time of 21 seconds is required. The effective measuring time, the time spent on measurements that are used, is three seconds.



It is possible to write a small program to measure the peak intensity as shown in the following example:

```
C = ASS 2        assemble program 2
A = ROF          recorder off
A = POF          printhead disabled
A = IAN -0.6     increment 2θ angle by -0.6°
A = SPE 1        set the scan speed to 1° 2θ per second
A = INT 0.05     set the sampling interval to 0.05 seconds
A = CSP 1        perform a continuous scan with peak labelling
A = /            end of program
```

This program differs from the search routine in that it does not return to the detected peak. To return to the peak in the search routine is useful for the following reasons:

- It is possible to measure and step from the peak.
- The peak shift caused by the surface height changes as 2θ alters.
- The diffractometer angle readout can be adapted to suit a particular class of samples, as prepared with a given error. This is done using the search routine together with the correct 2θ angle command.

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5.1 INTRODUCTION

This chapter introduces data manipulation, and relates closely to the chapter on programming. The results of the different routines are detailed, together with information on the presentation of this data and the units it is produced in. Measurements are subdivided into three distinct categories:

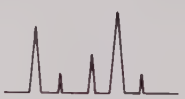
- Measurements around an angle.
- Scans.
- Measurements around a peak.

Measurements about an angle are performed using the measure intensity routine. This provides results in counts and intensity. It is important to remember that counts are always expressed in counts per second on the PW1710. Failure to allow for this while performing calculations can compromise the results obtained.

Scans can be effected in three different forms, continuous scans, continuous scans with peak labelling and step scans. The results of these scans are presented as diffractograms, standard output, counts and intensities.

Measurements around a peak are performed using three of the standard routines provided with the PW1710. Routines are provided to search for maximum intensity, to search for top intensity, and to measure area. Output from these modules is presented as diffractograms, standard output, counts and intensity.

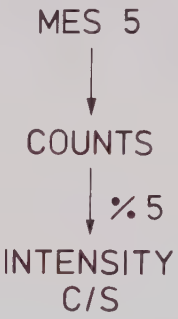
Diffractograms and standard output are given in a preprogrammed format. Diffractograms give the detected intensities in counts per second as a function of the diffractometer angle. Diffractograms are output on the recorder.



Standard output depends on the type of measurement. It can be angles, intensities or lattice spacing values, and appears on the device specified by the operator.

```
ANGLE  D  INTENSITY
26.670 3.3456 45023
```

Counts and intensity equate to one another on the PW1710. When a measurement is made it is necessary to specify the recording time. The number of pulses is counted and converted into intensity by dividing the value obtained by the measuring time. This useful value is automatically calculated by the system.



5.2 BASIC REGISTERS

This section introduces the basic concepts involved in the use of registers and details some basic procedures to gain access to the information stored in them.

All values used by the system are held in the system memory in storage areas known as registers. There is always a minimum of three registers reserved on the PW1710.

- A register to store counts.
- A register to store the intensity.
- The accumulator which is used to hold data to be manipulated.

After a measurement the counts and intensity registers hold the counts and intensity, respectively. The intensity value is also copied into the accumulator to allow processing of the measurement result.

COUNTS = pulses counted during a period

INTENSITY = counts per second

The values held in these registers remain available until the next measurement operation starts. If the values obtained are to be processed it must be done before starting another measurement operation.

There is a specific command to print the contents of the accumulator or any of the registers. The syntax of this command is detailed in the chapter on output. The system incorporates a further command to specify the number of decimal places to be printed when outputting the contents of the accumulator, or any of the registers.

The contents of the counts and the intensity registers can be displayed for viewing after any measurement operation. The values must be copied into the accumulator using the appropriate commands. Copying a value from a register, into the accumulator, doees not alter the value in the original register. Thus the same value can be used several times for different purposes, although new values will be placed in the registers when another measurement is performed. The commands used to manipulate and view data in the basic registers are individually detailed in the following paragraphs.

PTR n This command is used to print the contents of the register specified by the number "n". Entry of "0" specifies the contents of the accumulator.

DEC x This command is used to specify the number of decimals to be printed when using the print command to print the contents of a register. The allowed range for "x" is from zero to seven characters for output after the decimal point. There can be up to ten characters in a value, including decimal points and minus symbols.

RCT This command copies the contents of the counts register into the accumulator.

RIN This command copies the contents of the intensity register into the accumulator.

The following example demonstrates use of a series of commands which relate to the different registers.

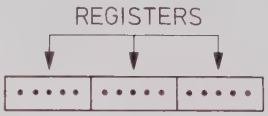
SAN a	set start angle
MES 10	measure for 10 seconds
DEC 0	set output to omit values after decimal point (the argument with this command can be in the range 1 to 7)
PTR 0	print the contents of the accumulator
DEC 2	set output to print two values after the decimal point when the print command is used
PTR 0	print the contents of the accumulator again (printed with two decimal places)
DEC 0	set output to omit values after decimal point
RCT	copy the contents of the counts register into the accumulator
PTR 0	print the counts value stored in the accumulator by the last command
RIN	copy the contents of the intensity register into the accumulator
PTR 0	print the intensity value stored in the accumulator by the last command

Counts and intensity remain available until the next measurement starts.

5.3 ACCUMULATOR

The accumulator is the working register. All data processing is carried out on information held in this location. Data processing refers to all arithmetic and logical operations as in the following list:

- Addition.
- Subtraction.
- Multiplication.
- Division.
- Square root.



The following paragraphs detail the commands used to perform data processing operations on the contents of the accumulator. In all cases the old value in the accumulator is replaced by the product of the specified operation.

ADD r Add the contents of register "r" to the contents of the accumulator ("r" can be from one to 255 as detailed in the section on supplementary registers).

SUB r Subtract the contents of register "r" from the contents of the accumulator.

MUL r Multiply the contents of the accumulator by the contents of register "r".

DIV r Divide the contents of the accumulator by the contents of register "r".

ADC v Add the value "v", supplied as an argument with the command, to the contents of the accumulator.

SUC v Subtract the value "v", supplied as an argument with the command, from the contents of the accumulator.

MUC v Multiply the contents of the accumulator by the value "v", which is given as an argument with the command.

DVC v Divide the contents of the accumulator by the value "v", which is given as an argument with the command.

SQR Calculates the square root of the contents of the accumulator.

The following example demonstrates a number of simple data processing steps:

```
RCT       this recalls the counts value to the accumulator
PTR 0     this prints the number of counts with the number of
          decimals specified by the last use of the relevant
          command
DVC 2     this divides the contents of the accumulator by two
PTR 0     this prints half the number of counts
MUC 8     this multiplies the contents of the accumulator, half
          the number of counts, by two
PTR 0     this prints the contents of the accumulator, half the
          number of counts multiplied by eight
```

Similarly the other processing functions can also be used as:

ADC 3000	add 3000 to the contents of the accumulator
PTR 0	print the contents of the accumulator
SUC 7000	subtract 7000 from the contents of the accumulator
PTR 0	print the contents of the accumulator
SQR	get the square root of the contents of the accumulator
DEC 3	use three figures after the decimal point when printing values
PTR 0	print the contents of the accumulator, with three decimal places having specified this in the previous command

The values in the accumulator do not have to be integers, fractions expressed as decimals are permitted. It is important to remember that failure to specify the appropriate number of decimals for output with the print command may result in the output of values which appear to be integers.

Having entered all the commands and processed the counts value it is still possible to recall it, as it was stored in the counts register.

5.4 SUPPLEMENTARY REGISTERS

The previous section introduced a number of simple processing operations. It is apparent that a facility to store information is necessary. The PW1710 has a facility whereby a number of extra registers can be made available. These registers are made available in the memory by declaring them using the register declaration command. The values in registers are always maintained until the register is cleared or overwritten. It is important to remember that all data processing is performed in the accumulator. The commands used with these registers are detailed in the following paragraphs.

NRG r This command is used to reserve "r" registers. Up to 255 registers are available for declaration in this manner. These registers are identified by numbers, and the number "0" is always assigned to the accumulator. If this command is used it is vital to remember that all currently stored values are cleared.

STR r This command is used to copy the contents of the accumulator into the register specified by "r". The accumulator still contains the same value after entry of this command.

PTR r This command is used to print the contents of register "r". Once again printing the contents of a register does not cause any change.

CLR r This command is used to clear the contents of register "r", replacing the previous value with zero.

RCL r This command is used to recall the contents of register "r" and write them into the accumulator.

The following paragraphs are devoted to an example of the use of registers for a number of simple processing operations.

MES 10	measure for ten seconds
PTR 0	print the intensity value stored in the accumulator
STR 4	store the intensity in register "4"
PTR 0	print the contents of the accumulator, which still holds the intensity measurement
CLR 0	clear the accumulator
PTR 0	print the value in the accumulator, which was cleared to zero by the previous command
PTR 4	print the contents of register "4", which holds the intensity value previously stored there
MES 7	measure for seven seconds
PTR 0	print the contents of the accumulator, which holds the intensity obtained during the seven second measurement
PTR 4	print the contents of register "4" which contains the intensity value obtained during the ten second measurement
DIV 4	divide the intensity value obtained during the seven second measurement by the value obtained during the ten second measurement
PTR 0	print the contents of the accumulator, the ratio between the two measurements
PTR 4	print the contents of register "4", the intensity obtained during the ten second measurement

5.5 APPLICATIONS

This section describes some of the applications that the PW1710 can be used for. The following facilities are particularly useful:

- Successive measurements can be added together and a background can be subtracted.
- Multiplication and division can be used to scale results:
  - Multiplication for concentration calculations.
  - Division to calculate a ratio between two values.

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6.1 INTRODUCTION

This chapter details the commands used to format data, specify where it is to be output, and how to control that output. Certain output is automatically formatted and output by the PW1710, and details on this fixed output are also provided in this chapter. The system has also got facilities to incorporate text and other information for output with results. These facilities allow the user to select direct output, or output after processing the measured data. All output must be programmed (refer to the chapter on programming) as it is only possible to output the contents of registers on an individual basis by direct command entry.

6.2 DATA VALUES OUTPUT

Data values are printed with a field width of ten characters including the sign, if negative, and the decimal point. Leading spaces are added to make up the ten characters. The number of decimal places displayed depends on the last specification with the control command.

If the value is longer than ten characters a series of asterisks is displayed instead. If this occurs it is necessary to re-specify the number of decimals to be displayed, and if necessary, divide the value by a multiple of ten to reduce the number of characters to the maximum permissible. Details on how to print or display the values are provided in the following sections.

6.3 PRINTED OUTPUT

Text consists of alphanumeric characters, that is the letters, numbers and symbols on a normal typewriter keyboard. Output is normally produced by programs as it is only possible to print individual values when exercising step-by-step control from the front panel or the operator´s terminal.

BFR r This command places the contents of register "r" in the output buffer with the number of decimal digits depending on the last specification.

PTR r This command stores the contents of register "r" in the output buffer and prints it. Once again the same rules apply for specification of the number of characters after the decimal point.

BFA xxx This command stores the characters "xxx" in the output buffer. The "xxx" characters can be any alphanumeric characters, including spaces, and a maximum of 16 characters are allowed.

PTA xxx This command stores the characters "xxx" in the output buffer, and then prints them. The "xxx" characters can be any alphanumeric characters, including spaces, and a maximum of 16 characters are allowed.

HED This command initiates printout of the heading, on terminal(s) depending on the output configuration specified. Specification of the output configuration is by means of the following four commands. They have already been referred to in chapter 2 but are repeated here for convenience.

DON This command is used to turn dual output ON. When the system includes two output devices simultaneous output to both can be specified using this command.

DOF This command is used to turn the dual output, selected by the previous command, OFF.

PON This command is used to enable the printhead on the recorder, if a suitable recorder is fitted.

POF This command is used to turn recorder printing OFF. This command is used to reverse the effect of the previous command if a suitable recorder is fitted.

6.4 DISPLAYED OUTPUT

DPL x This command is used to initiate a readout display, on the left hand side. The actual output depends on the entry made for "x". The different entry options are as follow:

- G for the 2θ goniometer angle.
- X for the scaler, counts or counts per second.
- D for the lattice spacing value.
- P for the program number.
- S for the sample number.
- C for the command.

- W for the wait condition. This can be any of the following:
  - . 1 for the goniometer.
  - . 2 for the timer.
  - . 3 for the timer or goniometer.
  - . 4 for terminal input.
  - . 5 for terminal output.
  - . 6 for second output device.
  - . 7 for the PM8210 transfer.
  - . 8 for the PM8210 printhead.
  - . 9 for the PM8202 line feed.
  - . 10 for delay after <CR>.
  - . 11 for echo input.
  - . 12 for the sample changer.
  - . 13 for input from a cassette recorder.
  - . 14 for input to continue after a halt, or because the system has been placed in the wait mode.
- T for the value of θ.
- F for the fluorescence scaler.
- B for the contents of the output buffer.
- O to cancel the display.

DPR x This command is used to initiate a readout display, on the right hand side. The actual output depends on the entry made for "x". The different entry options are as follow:

- G for the 2θ goniometer angle.
- X for the scaler, counts or counts per second.
- D for the lattice spacing value.
- P for the program number.
- S for the sample number.
- C for the command.
- W for the wait condition. This can be any of the following:
  - . 1 for the goniometer.
  - . 2 for the timer.
  - . 3 for the timer or goniometer.
  - . 4 for terminal input.
  - . 5 for terminal output.
  - . 6 for second output device.
  - . 7 for the PM8210 transfer.
  - . 8 for the PM8210 printhead.
  - . 9 for the PM8202 line feed.
  - . 10 for delay after <CR>.
  - . 11 for echo input.
  - . 12 for the sample changer.
  - . 13 for input from a cassette recorder.
  - . 14 for input to continue after a halt, or because the system has been placed in the wait mode.
- T for the value of θ.
- F for the fluorescence scaler.
- B for the contents of the output buffer.
- O to cancel the display.

WTD This command is used to introduce a two second wait. It is normally used after writing to the buffer to allow time to read the information displayed on the readout.

HLT This command is used to stop operations, within a program. Entry of <CR>, by the operator causes the program to continue.

6.5 DISPLAYED MESSAGES

The buffer display facility is intended for data output, but it can also be used to show user-made messages. For example it is possible to write a short program to display a message to display "FINISHED", or similar text. This program can then be called at the end of a long measurement routine. It is also possible to cause the display to flash, drawing attention and eliminating the need to follow long measurement operations closely. The following example demonstrates such a program.

```
C = ASS 191          assemble program 191
A = DPR 0            right hand side of readout display OFF
A = BFA FINISHED     store "FINISHED" in output buffer
A = WTD              wait 2 seconds after writing to buffer in previous
                     line
A = PTA<blank>       write a blank space to the buffer and print it
A = WTD              wait two seconds
A = /                end of program
C = ASS 190          assemble program 190
A = REP 191 0        repeat program 191 indefinitely
A = /                end of program
C = CLP 190          call program 190
```

As can be seen the flashing effect is achieved by repetition of the output. It is not suitable for use when operatuing in remote mode with a printer as it will produce continuous print until the program is halted. Similar routines can be written to prompt operators with appropriate messages.

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7.1 INTRODUCTION

This chapter introduces programming and provides examples of programs in order to familiarise users with the simple procedures required. Programs are made up of a series of steps, each step being the execution of one of the commands detailed in this manual. Programs can be stored and recalled, eliminating the necessity to repeat a sequence of commands by direct entry. Normal programs break up into the following main steps:

- Preparation of the instrument settings.
- Measuring.
- Data handling.
- Output.

Each of these steps may require one or more instructions. Preparation involves setting parameters, such as goniometer speed, counting time, recorder control and so on. Measuring includes the setting of angles and initiation of a measuring action. Data handling requires the performance of calculations, formatting and storage of results. Output refers to the printing of results with suitable text.

Programs can be called by their numbers. When called all the instructions will be executed, in the order that they occur in the program. Using programs provides other advantages in that actions can be repeated precisely, and there is no danger of omitting a step within a procedure.

The system already includes some ready-made parts with can be called by a single instruction, such as the search for maximum intensity routine. It still remains necessary to set certain parameters however. The parameters which must be set when using the routines supplied with the system are detailed in the earlier chapters.

7.2 PROGRAMMING

This section details the approach to be adopted when writing and entering programs. Programming breaks down into the following steps:

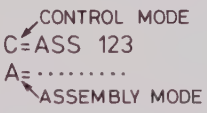
- Definition of the job to be done.
- Breaking the job down into steps, in appropriate order.
- Entering the instructions into the system memory. This step is known as program assembly.

ASS p This command is used to place the system in ASSEMBLY mode, ready for entry of the actual steps making up a program. The number "p" refers to the number which is used for that program from then on. When the system is placed in ASSEMBLY mode the prompt:

A =

is displayed to the operator, to show that the system is ready for program entry. The ASSEMBLY and BUSY lamps on the front panel are illuminated and the readout on the front panel displays the letter "A" and a value on the right hand side to indicate the amount of memory available on the system.

ASSEMBLY MODE



Entry of programs is then accomplished by entry of the appropriate commands, each one followed by <CR> or the ENTER push-button, depending on whether the system is in REMOTE or FRONT PANEL mode. After entry of each step the ASSEMBLY mode prompt is displayed to indicate that the system is ready to accept the next instruction.

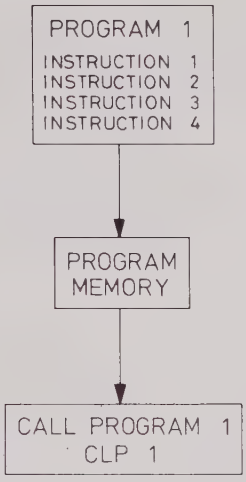
When all the instructions have been entered exit from ASSEMBLY mode by entry of a "/" followed by <CR> if operating in REMOTE mode. If operatiing in FRONT PANEL mode press the STOP push-button. The system then displays the normal command entry prompt, and the ASSEMBLY and BUSY lamps go OFF.

CLP p When a program has been successfully entered, and the system is back in COMMAND mode this instruction is used to call and execute it. The "p" refers to the program identification number given with the command to place the system in ASSEMBLY mode. It is not necessary to enter the full command as entry of a program number is sufficient.

If the the call program command is entered within a program, calling itself, the program will be executed five times and the error message:

** 6 STACK OVERFLOW

will be displayed. This should not be confused with calls for other programs which can be included. Details on such calls are given in the section on nesting programs. A short example of a program is provided with characters enclosed in chevrons, < >, representing keys inscribed with the enclosed characters.



REP p n This command performs the same function as the call and execute program command. The difference is that it is used for multiple executions of a program instead of a single execution. The "p" refers to the program number and the "n" specifies the number of times it is to be executed.

The following example demonstrates entry of a single program, with an explanation of each command provided beside it.

C =ASS 5<CR>	call ASSEMBLY mode to enter program 5, when this command is entered the BUSY and ASSEMBLY lamps on the front panel are lit, and the readout on the front panel displays a message indicating the amount of free memory
A =MES 10<CR>	perform a measurement for ten seconds
C =PTR 0<CR>	print the result of the ten second measurement
C =STR 4<CR>	store the result in register 4
C =PTR 0<CR>	print the contents of the accumulator which still holds the result of the ten second measurement
C =CLR 0<CR>	clear the accumulator replacing the contents with zero
C =PTR 0<CR>	print the contents of the accumulator
C =PTR 4<CR>	print the contents of register 4, the result of the ten second measurement
C =MES 7<CR>	perform a measurement for seven seconds
C =PTR 0<CR>	print the result of the seven second measurement
C =PTR 4<CR>	print the contents of register 4
C =DIV 4<CR>	divide the contents of the accumulator by the contents of register 4
C =PTR 0<CR>	print the contents of the accumulator, the ratio between the results from the seven and ten second measurements
C =PTR 4<CR>	print the contents of register 4, the result of the ten second measurement
C =RCL 4<CR>	load the contents of register 4 into the accumulator
C =PTR 0<CR>	print the contents of register 4
C =/<CR>	terminate program assembly, the command prompt is displayed and the ASSEMBLY and BUSY lamps go out
A =CLP 5<CR>	load and execute program 5

PRP p This command is used to output a list of program "p". Leaving the "p" out causes all the programs to be listed.

HLT This is a particularly useful command which can be incorporated in programs for a variety of reasons. When a program reaches the command it stops and waits for entry of a <CR> if it is in REMOTE mode, or use of the ENTER key if it is in FRONT PANEL mode. This allows programs to be written with a halt to check on a value before continuing. This saves unnecessary loss of time carrying out measurements on unsuitable samples.

7.3 PROGRAM NUMBER SELECTION

Program numbers can be in the range 1 to 255. There is a program "0" but this is reserved for use when switching the system ON. A special instruction is provided to list the numbers that have already been used. The instructions used when numbering programs are detailed in the following paragraphs.

PMM This command is used to print a list of all the programs currently in the memory. An example of this command is provided below with typical output.

```
C = PMM<CR>
  0   1   2   3   4   9  10  17  18  19  20  21  41  42  43
 44  47  60  61  66 100 120 121 122 123 124 125 126 127 128
129 130 131 180 181 182 183 184 185 186 187 188 189 190 191
192 193 194 196 197 198 199 200 201 210 211 217 220 221 222
223 224 225 226 227 228 229 230 231 232 233 234 235 236 237
238 239 240 241 242 243 244 245 249 250 252 253 254 255
NR OF REGISTERS=   1
FREE SPACE 1599 BYTES
```

In the above example numbers 5, 6, 7, and 8 are omitted because they are not in use. It is advisable to assign program numbers in blocks, each block containing related jobs, possibly produced by modifying similar programs within the same block.

Ideally multiples of ten should be reserved for main programs and variations numbered within that block, for example variations on program 50 should have related numbers in the same family, 51, 52, and 53. Programs to be used in combination should also receive related numbers. Further detail on this type of operation is given in the section on nesting programs.

Text and heading output routines should be given high numbers like 230 to 255. When using a given program it is a good idea to print it, before the output from the actual run. This allows rapid identification of the stage in the process at which events occurred when studying the results. It is also advisable to take a list of the parameter settings.

7.4 PROGRAM MODIFICATION

It is often necessary to modify programs for a variety of different reasons. Mistakes may have been made when entering them originally, or it may be necessary to change a program by extending the functions to be carried out.

Alternatively it may just be necessary to change the preprogrammed angles, measuring times or ranges. It is recommended that the following guidelines are used when modifying programs:

- Do not modify other people's programs as this could lead to major problems if they are unaware of it.
- Do not modify standard programs, make variations instead.

If the program to be modified is not a main program, does not belong to someone else, and only requires correction of lines already inserted, or modification of some of the parameters, it is easy to overwrite the lines to be changed.

It is also easy to insert instructions between previous instructions, or add extra instructions at the beginning or end. Instructions to be removed from programs must be individually deleted. When changing the sequence of a series of instructions it is necessary to insert and delete lines at the appropriate places. There is also a command to delete whole programs.

INS — This is the INSERTION mode entry command. Entry of this command places the system in INSERT mode, and the new line or lines entered are placed immediately in front of the line displayed when the command was entered. Entry of new lines is effected in the same way as initial line entry in ASSEMBLY mode when entering a program for the first time.

INSERT
"INS"

DEL — Entry of this command against any line in a program, when the system is in ASSEMBLY mode, results in removal of that line. An example is provided in the following example.

DELETE
"DEL"

DLP p — This is the command used to delete a program which is no longer required. The "p" refers to the number assigned to the program when it was originally entered on the system.

DELETE A PROGRAM
DLP 123

The following example demonstrates the different steps that are used to change programs. It uses program "5", as entered in the previous example. In each case the screen text appears as shown, as far as the "=" sign on each line, with the program number displayed beside the first line. The text explaining the function of each command has been omitted as the command functions have already been explained for this example. Entry of a <CR> causes the display of the next line and leaves the displayed command as it is. If the command is to be changed it is simply a matter of entering the new command, followed by <CR>.

```
    C =ASS 5<CR>          this entry causes the display shown on the following
                          line, the BUSY and ASSEMBLY lamps on the front panel
                          are lit, the readout on the front panel displays the
                          amount of memory available, and the first command is
                          displayed as shown on the following line
5   :MES 10=<CR>          this leaves the first line of program 5 as it was
     PTR 0=<CR>           once again this line is left unchanged
     STR 4=<CR>           store the accumulator contents in register 4
     PTR 0=<CR>           print the contents of the accumulator
     CLR 0=<CR>           put the value zero in the accumulator
     PTR 0=PTR 4<CR>      this command now prints the contents of register 4 not
                          register 0
     PTR 4=PTR0<CR>       this command now prints the contents of register 0, not
                          register 4
     MES 7=<CR>           measure at the current angle, for seven seconds in 0.05
                          second intervals
     PTR 0=<CR>           print the contents of the accumulator
     PTR 4=DEL<CR>        this entry deletes the old command to print the
                          contents of register 4
     DIV 4=/<CR>          this exits from ASSEMBLY mode, implementing the changes
                          entered up to this point and leaving the rest of the
                          program as originally entered
     A =CLP 5<CR>         load and execute program 5
```

It is very important to remember that exiting from the assembly mode in the middle of a program has no effect on the rest of the program. Listing program "5" shows that the instructions from the point of exit are still the same. If it is necessary to remove a series of commands from a particular point to the end of a program each command must be individually deleted. Once again be very careful not to put a call for program "5" in before ensuring that the system has left the assembly mode, as indicated by display of the command prompt.

It may be necessary to add extra instructions between those previously entered in a program. The PW1710 has a special facility to do this. The INSERTION mode allows simple addition of new commands. The following example uses the same program again, and demonstrates the addition of an extra command to multiply the result obtained during the seven second measurement, before calculating the ratio between the two measurements.

```
C =ASS 5<CR>          this entry causes the display shown on the following
                      line, the BUSY and ASSEMBLY lamps on the front panel
                      are lit, and the readout on the front panel displays
                      the amount of memory available and the first command is
                      displayed as shown on the following line
5  :MES 10=<CR>       this leaves the first line of program 5 as it was
                      previously
   PTR 0=<CR>         once again this line is left unchanged
   STR 4=<CR>
   PTR 0=<CR>
   CLR 0=<CR>
   PTR 0=PTR 4<CR>this command now prints the contents of register 4 not
                      register 0
   PTR 4=PTR 0<CR>this command now prints the contents of register 0
   MES 7=<CR>
   PTR 0=<CR>
   PTR 4=DEL<CR>      this entry deletes the old command to print the
                      contents of register 4
   DIV 4=INS<CR>      this places the system in the insertion mode, and the
                      insert prompt is displayed as shown on the next line,
                      ready for entry of new commands which are placed in
                      front of the last command displayed
   I =MUC 4<CR>       this multiplies the result of the seven second
                      measurement by 4
   I =/<CR>           this exits from the INSERTION mode and returns the
                      system to normal ASSEMBLY mode, further instructions
                      can be entered first if required
   PTR 0=/<CR>        this terminates modification of program 5 and returns
                      the system to COMMAND mode, leaving all the commands
                      between the exit point and the end of the program as
                      they were previously
   A =CLP 5<CR>       load and execute program 5
```

To sum up the PW1710 has a number of operational modes which can be used to perform different functions. COMMAND mode allows direct control of the system on a single step command entry basis, and also allows the loading and execution of programs. The ASSEMBLY mode allows entry of programs and deletion and modification of commands. The INSERTION mode allows insertion of additional instructions. Lists of programs from here on do not incorporate <CR> symbols, as they are illustrating command functions, not the actual operator entry.

7.5 PROGRAM MEMORY

When the system is placed in ASSEMBLY mode all the commands entered are automatically stored in a special area of memory reserved for that purpose. These stored programs are maintained in memory as long as power is available. The system includes a battery power supply to maintain the memory contents for six or seven weeks with the power switched OFF. The memory has a fixed size, which is expressed in bytes. The PW1710 has approximately 1,500 bytes of memory available to store programs.

When a program is being assembled the readout on the front panel displays the amount of memory available, in bytes, on the right hand side. As a program is entered the amount of space decreases and the readout display is reduced accordingly. Instructions require a varying amount of space. This is clearly shown on the readout, with varying decrements, depending on the command entered. When a register is declared it uses four bytes, and the command occupies two bytes. The space used by registers is the number of registers last declared using the register declaration command.

Text strings use a lot of memory. Therefore it is better to try and limit the length of strings to be used in programs, or alternatively to write separate programs to perform all the text printing functions. These separate routines can then be called to perform their functions, within other programs. This is known as nesting and is detailed later in this chapter. If the memory capacity is exceeded when assembling programs the system displays the folowing error message:

** 3 MEMORY OVERFLOW

If this message is displayed it is necessary to delete some of the programs already on the system before any further programming can be carried out. The facility to record programs on cassette tapes is particularly useful. It provides extra storage when the system memory does not have sufficient capacity to store all the user's programs.

It is always possible to check on the amount of memory left available on the system by listing the programs, as the listing incorporates a message saying how much memory is still available. It is also advisable to try to keep some memory free. If the memory is fully occupied the error message:

** 3 MEMORY OVERFLOW

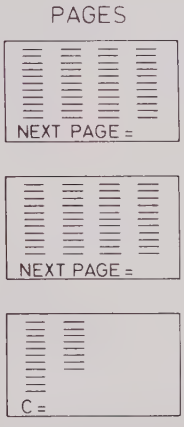
is displayed when a program declaring extra registers is called and run.

7.6 PROGRAM LISTING

The command to obtain a listing of any one program, or all the programs is detailed previously. This listing can be obtained in a number of different forms, depending on the type of output specified. Further information on output specification is provided in the previous chapter. The different types of output are described here to help interpret them. The different options are:

- On the operator's terminal.
- On a second terminal when dual output is specified.
- On the front panel readout by entering ASSEMBLY mode and stepping through the instructions one by one. It is important to remember to use the STOP push-button to exit ASSEMBLY mode if this method is used.
- On a printing recorder, for example the PM 8210. In this case it is important to remember that the commands are listed in reverse order, with the last command first, and so on.

When the listing is output to a terminal it is produced in pages, each of which can have up to 255 lines, in up to four columns. The number of lines and columns to be printed is selected using the print specification command explained below. Each column is 25 characters wide. This is necessary to reserve space for long instructions such as the command used to place text strings in the output buffer which can require up to 20 places. The remaining five character spaces are reserved for program numbers and column separation.



NLC 1 c This command is used to specify the number of lines and columns when printing. The "1" refers to the number of lines which can be in the range one to 255. The "c" refers to the number of columns, which can be in the range one to four. When selecting the number of lines to be printed consider the size of the paper sheets being used for the output, or the screen size if printing on the screen. For example the normal terminal size permits display of 23 lines using three columns.

When each page is displayed the system waits for a <CR> before carrying on to print the next page. It is recommended that an up to date list of all, the programs and their listings is maintained near the equipment for the convenience of all the users.

7.7 NESTING PROGRAMS

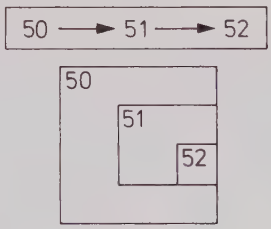
The command to call and run programs has been explained earlier. This section explains other uses for this command. Previous reference has been made to the fact that this command causes error messages, if the program calls itself. It is possible to call other programs from a program however. This facility can be put to a number of uses, although there are a number of potential areas of confusion for inexperienced users.

Nesting can be used to call a group of actions, a program, using only one instruction, thus saving re-entry of a sequence of commands already in memory. The logical extension of this is that small modular programs for different common tasks can be written, and called by other programs, providing a structured approach.

Programs called by other programs can in turn call further programs, although the number of calls that can be nested in this manner is limited to six. If this limit is exceeded the error message:

** 6 STACK OVERFLOW

is displayed. The main disadvantage encountered when using nesting is that it is not always easy to interpret what precisely is being done. It can also lead to errors because one or more of the programs called may have been changed, thus introducing possible errors.



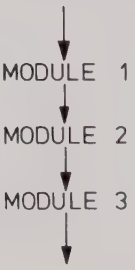
NESTING

It is recommended that nesting is used for the following applications:

- When using the same sequence of instructions more than once within a job.
- When a series of jobs has to be done.
- For text output.
- When decisions as to the procedure to be used next depend on the results obtained so far.

```
C=ASS 70
A=CLP 71
A=CLP 72
A=CLP 73
A=/
```

When using nesting it is important to approach the system logically. Divide the actions to be performed into logical modules, and enter each module as a separate program. When all the modules have been put into memory it is a simple matter to write one final program consisting of a series of calls for each of the modules, in the required order. Where actions have to be repeated this also allows recall of the same program several times, saving memory.



When nesting is used properly it has the following advantages:

- The function of a properly structured modular system can be easy to interpret.
- If an individual instruction must be changed it is only necessary to modify the module it occurs in.
- Modules can be used several times in different experiments where there is a common procedure.
- A module can be repeated several times, within a job, using the repeat command.
- Modules can be inserted and removed from jobs by simply deleting or inserting calls in the control program, the program made up of calls for each of the modules.
- The order of text presentation, by modules, can easily be changed.

The following advice should prove useful when using nesting:

- When learning to program as it is better to concentrate on producing simpler programs and avoiding complicated programming tricks.
- Avoid making the modules too small as it becomes very difficult to interpret what the overall task is and there is a danger that individual, seemingly unimportant routines may be deleted, causing the whole job to go wrong.
- Do not use nesting for important basic programs.
- Do not use modules written by other people in nested jobs as they may change them.

The following paragraphs explain why it is advisable to use nested routines for production of text, and demonstrate the type of procedure that should be adopted to ensure satisfactory layout. Layout is important when text is being located around values, for example the heading on a table. In this case a lot of spaces are normally required. Putting large numbers of spaces in the program, using the store characters in the output buffer command, uses a lot of memory. The following example demonstrates a typical pair of programs for printing purposes.

```
C =ASS 255<CR>
A =BFA <CR>
A =/<CR>

C =ASS 254<CR>
A =PTA<LF><CR>
A =/<CR>
```

In program "254" the symbol <LF> refers to the line feed key on the terminal keyboard. This key may be identified by the words LINE FEED or by a vertical pointer, depending on the type of terminal in use. Program "255" inserts a space in the output buffer and the paper is raised one line by program "254".

TITLE

TABLE

COLUMN1	COL.2	COL.3
1	XXX	X.X
2	XX	X.X
3	XXXX	X.X

CONCLUSION

REP p n This command performs the same function as the call and execute program command. The difference is that it is used for multiple executions of a program instead of a single execution. The "p" refers to the program number and the "n" specifies the number of times it is to be executed.

Using the above command to call the two example programs allows formatting of data. Spacing between headings for example, is inserted, by using example program "255" to insert the required number of spaces in the output buffer. Program "254" allows the insertion of line feeds to space headings apart from their tables and so on. Similarly separating lines between listings can be produced with a simple program like program "252".

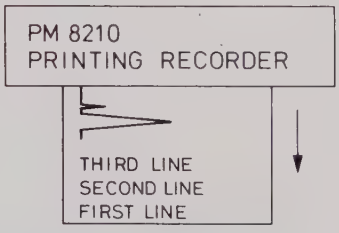
```
C =ASS 252<CR>
A =PTA *<CR>
A =/<CR>C =
```

Use of the call and multiple execution command allows use of this routine to insert a line of asterisks between two separate listings. For example entry of the command:

```
REP 252 65<CR>
```

followed by the print output buffer command produces a suitable separation line for insertion between two listings. It is also advisable to use a similar procedure if the same line is to be printed several times during a job.

When printing is being produced on a printing recorder it is useful to print each line in a separate routine. The printing recorder produces text with the line order reversed, but it is a simple matter to reverse the order in which lines are output, thus putting it back in the required order. Programs "245" and "244" demonstrate this. Program "244" is the reverse of "245", thus using "244" when outputting to a printing recorder produces the same output as "255" on a normal printer.



```
C =ASS 245<CR>
A =PON<CR>
A =POF<CR>
A =CLP 246<CR>
A =CLP 247<CR>
A =CLP 248<CR>
A =CLP 249<CR>
A =POF<CR>
A =/<CR>

C =ASS 244<CR>
A =PON<CR>
A =CLP 249<CR>
A =CLP 248<CR>
A =CLP 247<CR>
A =CLP 246<CR>
A =POF<CR>
A =/<CR>
```

The system also incorporates a number of other commands which enhance flexibility. The following paragraphs detail these commands and describe the type of application they have been designed for.

CPO p This command is used to call and run a program, when the value held in the accumulator is greater than or equal to zero. It is normally used within a program, calling another program when a certain condition exists. An example of a typical program using this command and the command to call a program when the value held in the accumulator is less than zero is provided below.

CNE p This command is used to call and run a program when the value held in the accumulator is less than zero. As with the previous command it is normally used to call a program from within another one when a certain condition prevails. Use of this command is demonstrated in the following example.

MXN 10	this specifies that there are to be ten measuring points each side of the specified angle
MXS 0.010	this specifies the step size between each of the measuring points
MXT 0.10	this specifies that each point is to be measured for 0.10 seconds
SAN 26.650	this specifies the angle 26.650^{o} to be used as the start angle and ten measurements will then be taken each side of it
MAX	this calls the maximum intensity measurement routine
SUC 15000	this subtracts 15,000 from the maximum intensity value placed in the accumulator by the maximum intensity measurement routine
CPO 18	this calls and runs program 18 if the value held in the accumulator is greater than or equal to zero
CNE 19	this calls and runs program 19 if the value held in the accumulator is less than 19

7.8 EXAMPLE APPLICATION

The following example is provided to demonstrate a typical application using the PW1710. In this example the sample is approximately 0.1 mm too high. As detailed previously this induces an error, about 0.06° 2θ.

```
CAN 30.00      will correct the readout angle to 30.00°
MAX            calls and executes the maximum intensity measurement
               routine around the corrected angle
```

For samples containing quartz the following calibration procedure is typical.

```
C = ASS 17     assemble program 17
A = MXN 10     take ten measuring points on each side of the angle
A = MXS 0.01   make a step of 0.01° 2θ between points
A = MXT 1      measure for one second at each point
A = SAN 26.65  set the 2θ angle to 26.65°
A = MAX        carry out the maximum intensity measurement routine
A = SUC 20000  subtract 20000 from the contents of the accumulator
A = CPO 18     call program 18 if the value of accumulator contents is
               greater than or equal to zero
A = CNE 19     call program 19 if the contents of the accumulator are
               less than zero
A = /          end of program 17


C = ASS 18     assemble program 18
A = CAN 26.670 correct readout angle to 26.670°
A = PTA RECALIBRATED store the message RECALIBRATED in the output
               buffer
A = /          end of program


C = ASS 19     assemble program 19
A = PTA TOO LOW INTENS stores the message TOO LOW INTENS in the output
               buffer
```

This set of three programs is coupled. Program "17" searches for the maximum intensity. Then it subtracts 20,000 from the measured value. When that intensity is more than 20000 the difference is positive, so program "18" will be called, resetting the readout angle. If the intensity is less than 20,000, program "19" will be executed instead. As can be seen printing the contents of the output buffer will then indicate what has happened. Running program "17" with the shutter closed will therefore store a message warning that the intensity was too low in the output buffer. This type of procedure would be adopted in situations where only high peak measurements were required.

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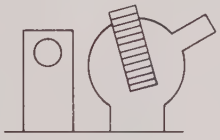
8.1 INTRODUCTION

This chapter details different options available with the PW1710 and explains the commands specific to their use. Options such as these are used to extend the system for specific applications. System design has been implemented to allow easy reconfiguration of the system with any of the options fitted. This design philosophy means that options can be purchased and added to the system after installation, as and when required.

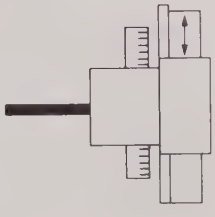
8.2 PW1170 SAMPLE CHANGER

The PW1170 is an automatic sample changer. The sample magazine can take up to 35 samples, and a separate loading platform is provided for priority or monitor samples. This sample changer is particularly suitable for use when sample shapes are not consistent. The following paragraphs detail the commands for the PW1710, which are specific to this option..

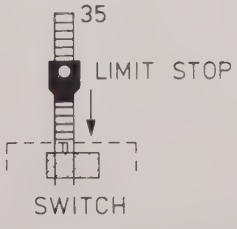
DPR S Display the sample number on the right side of the readout on the front panel. This refers to the magazine position, in other words it shows which sample is on the loading level.



MAG i Set the magazine to position "i". When the magazine is moving towards a position the sample number follows on the display. The acceptable values for "i" are from zero to 35, with both zero and one referring to the bottom position.



LSM This command is used to load a sample from the magazine. When a PM8203A recorder is fitted and has been enabled, the sample number is passed to the recorder and the paper is fed up ten mm. If the PM8210 recorder is fitted this does not happen.



It is possible to use the print heading command to produce a heading and separate measurements. The PM8210 uses five mm for each line of text. It is advisable to include a line feed after the heading to start recording on a one cm line, if a series of recordings are being carried out by a single program.

USM This command is used to unload a sample, back to the magazine.

The sample changer is designed for use when a series of similar samples are to be measured using the same procedure. A special command has been included on the system, for use in this situation. Basically this command allows specification of a series of magazines. The command is made up of two parts, which must be used together and can only be used within programs. All the measurement and calculation functions for the samples to be tested must be enclosed between these two parts.

SCH i j This is the first half of the repeated measurement command. The number "i" refers to the first sample, giving the position number containing the first sample. The "j" gives the position number containing the last sample for analysis. It is not necessary to have a sample in each position, although it is a waste of time to leave blank positions as the system still measures them.

NXS This is the second half of the repeated measurement command. If more samples are to be measured the system returns to the start of the enclosed sequence of commands and steps the sample changer to the next position.

Groups of samples to be measured in different ways can be placed in the magazine. It is then necessary to write routines for each of these blocks. Such routines can all be incorporated in one program, if required. It is advisable to write the measurement and calculation sections for each block in separate programs called by the main program. This is demonstrated in the following example program:

SCH 1 5	this specifies performance of the instructions between this start of the section and the end, moving the magazine to position "1" and then repeating the same procedure for positions "2" to "5"
CLP 10	this calls and runs program "10", which incorporates all the control commands necessary for measurement and result display
NXS	this is the second half of the first repeated measurement command
SCH 6 15	this is the start of the second repeated measurement routine
CLP 11	this is the program to perform the second measurement
NXS	this is the end of the repeated measurement routine
SCH 16 21	this is the start of the third repeated measurement routine
CLP 12	this calls and runs program "12", which incorporates all the control commands necessary for measurement and result display
NXS	this is the end of the repeated measurement routine
SCH 22 35	this is the start of the fourth repeated measurement routine
CLP 13	this calls and runs program "13", which incorporates the commands necessary for measurement and display
NXS	this is the end of the repeated measurement routine

There are three error messages that may be encountered when using the sample changer. They are as follow:

** 17 CHANGER ERROR

This message is displayed if a mechanical fault is detected or when the changer is not in the correct position. An example of what is meant by the incorrect position is provided by the case where the limit stop on the magazine transport has been triggered during run-up.

** 18 NO CHANGER

This message indicates that the sample changer is not connected.

** 19 SAMPLE IN GON

This message indicates that there is already a sample in the goniometer.

The system can be switched OFF with the sample changer in any position. When it is switched ON again the PW1710 calculates the position of the changer. Sometimes this calculation cannot be carried out, and the magazine is run down to the zero position to define the position. When running down in this manner the magazine position number on the display is not changed, but it is corrected after the system has been reset.

Always check the sample loading rod after switching ON, and enter the load sample from magazine or the unload sample to magazine command. It is possible to select a different position when a sample is loaded. Dual entry of the load or unload commands is ignored.

8.3 PW1774 SAMPLE SPINNER

The PW1774 sample spinner is designed for use with samples which do not have a homogeneous surface. They are spun at high speed thus providing measurements as if a homogeneous sample was used. When the PW1774 is used with a PW1775 sample changer the spinner automatically stops for loading and unloading operations.

SON This command is used to switch the PW1774 spinner ON.

SOF This command is used to switch the PW1774 spinner OFF.

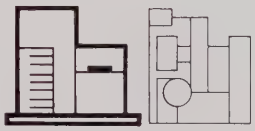
8.4 PW1775 SAMPLE CHANGER

The PW1775 is an automatic sample changer. Two sample magazines are available for use, a 21 position magazine for large samples, and a 42 position magazine. When using a PW1775 it is necessary to use one or other of the magazine options and the machine must be set accordingly.

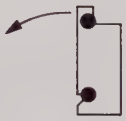
A separate loading platform is provided for priority or monitor samples. The PW1775 detects empty positions during a normal series of sample measurements, and ignores them when a normal program to measure a series is operating. It can also be used to measure variable numbers of samples, with one program using different measuring routines, with the changeover of routines on detection of an empty position. The following paragraphs detail the commands which are specific to the PW1775 option.

The sample changer is designed for use when a series of similar samples are to be measured using the same procedure. A special command has been included on the system for use in this situation, to save the time and effort required writing programs to specify the magazine number. Basically this command allows specification of a series of magazines. The command is made up of two parts, which must be used together and can only be used within programs. All the measurement and calculation functions for the samples to be tested must be enclosed between these two parts.

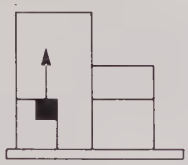
DPR S Display the sample number on the right side of the readout on the front panel. This refers to the magazine position, in other words it shows which sample is on the loading level.



MAG i Set the magazine to position "i". When the magazine is moving towards a position the sample number follows on the display. The acceptable values for "i" are from zero to 35, with both one and zero referring to the bottom position.

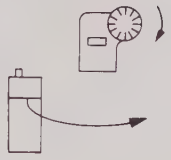


LSM This command is used to load a sample from the magazine. The goniometer shaft is automatically set to the angle required for loading, 2θ at 12 °.



When a PM8203A recorder is fitted, and has been enabled, the sample number is passed to the recorder and the paper is fed up ten mm. If the PM8210 recorder is fitted this does not happen.

It is possible to use the print heading command produce a heading and separate measurements. This recorder uses five mm for each line of text. It is advisable to include a line feed after the heading to start recording on a one cm line, if a series of recordings are being carried out by a single program.



USM This command is used to unload a sample, back to the magazine. The system automatically sets the goniometer shaft to the appropriate angle for unloading on entry of this command.

SCH i j This is the first half of the repeated measurement command. The number "i" refers to the first sample, giving the position number containing the first sample. The "j" gives the position number containing the last sample for analysis. It is not necessary to have a sample in each position although it is a waste of time to leave blank positions, as the system still measures them. A further facility allows specification of a search for the first position containing a sample, followed by repetition of the measurement procedure until an empty position is detected. This is done by specifying zero for both the "i" and "j" values. Repetition of this command allows routines similar to the example, with undefined length runs for each measurement routine. Variable length measurements can then be carried out by leaving positions empty in the magazine to define the start and end of each section.

NXS This is the second half of the repeated measurement command. If there are further samples to be measured the system then returns to the start of the enclosed sequence of commands and steps the sample changer to the next position.

Groups of samples to be measured in different ways can be placed in the magazine. It is then necessary to write routines for each of these blocks. Such routines can all be incorporated in one program, if required. It is advisable to write the measurement and calculation sections for each block in separate programs called by the main program. This is demonstrated in the following example program:

SCH 1 5	this specifies performance of the instructions between the start of the section and the end, moving the magazine to position "1" and then repeating the same procedure for positions "2" to "5"
CLP 10	this calls and runs program "10", which incorporates all the control commands necessary for measurement and result display
NXS	this is the second half of the first repeated measurement command
SCH 6 15	this is the start of the second repeated measurement routine
CLP 11	this is the program to perform the second measurement
NXS	this is the end of the repeated measurement routine
SCH 16 21	this is the start of the third repeated measurement routine
CLP 12	this calls and runs program "12", which incorporates all the control commands necessary for measurement and result display
NXS	this is the end of the repeated measurement routine

There are three error messages that may be encountered when using the sample changer. They are as follow:

** 17 CHANGER ERROR

This message is displayed if a mechanical fault is detected or when the changer is not in the correct position. Incorrect position refers to situations where the limit stop on the magazine transport has been triggered during run-up, or similar problems.

** 18 NO CHANGER

This message indicates that the sample changer is not connected.

** 19 SAMPLE IN GON

This message indicates that there is already a sample in the goniometer.

Do not use the load sample from magazine, the unload sample to magazine, or the set the magazine position command within repeated measurement routines. Similarly, do not use the single sample commands, detailed in the following paragraphs, within these routines.

The PW1775 incorporates facilities to load a sample from a single sample table, without using the magazine. The sample thickness must match the thickness used with the magazine option for which the system has been configured.

LSS This command is used to load a single sample. The system then sets the goniometer shaft for loading. When a PM8203A is fitted and has been enabled a signal of 42.5 scale divisions is recorded, during loading. Once loading has been carried out the shutter opens and the spinner, if fitted, rotates, if the appropriate command has been given to switch the spinner ON.

USS This is the reverse of the previous command and is used to unload single samples.

Single sample measurements may be required for a variety of reasons:

- A test sample.
- High priority samples.
- As a monitoring sample.
- To look at a particular level.
- To calculate a ratio.
- To provide a basis for comparisons.

8.5 PW1747 SEPARATE THETA DRIVE

This option is provided for measurements when working in the field of stress analysis. Fitting this option requires the use of some extra commands and they are detailed in this section. The operation of some other commands is no longer possible, for example the correct angle readout command cannot be used. The commands influenced and the new commands are as detailed in the following paragraphs.

STT a Set the 2θ goniometer angle to "a°".

ITT a Increment 2θ by "a°", with both positive and negative values permitted.

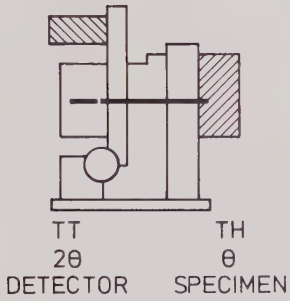
STH a Set θ to "a°".

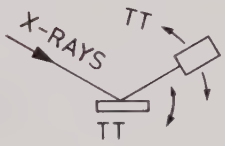
ITH a Increment θ by "a°" with both positive and negative values permitted.

CTT a Correct the 2θ readout at the angle "a°" in 0.005° steps.

CTH a Correct the θ readout at the angle "a°" in 0.005° steps.

PAR When a parameter listing is output values for θ and 2θ are displayed.





The commands to set the angle, to set the angle to twice the arcsine of the wavelength divided by twice the lattice spacing, and to increment the 2θ angle all produce coupled settings as detailed previously.

MOD x This command defines the scan mode with the values for "x" producing the following effects:

- "x" = 1 to move only the θ shaft.
- "x" = 2 to move only the 2θ shaft.
- "x" = 3 to move both shafts with the movement coupled in the ratio 1:2 for θ:2θ.

When operating in mode "1" all input and output is with reference to the θ angle, but when operating in modes "2" or "3" all input and output is with reference to 2θ.

It is possible to offset the θ value for use with the scanning routines, with the search for maximum intensity and the search top intensity around current angle and subtract background routines. It is important to remember that if this is done the θ value is no longer equal to half the 2θ value. Always check whether an offset has been introduced before using mode "3" with any of the above mentioned routines.

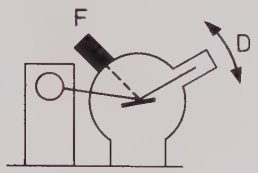
It is also essential to note the zero corrections after the mechanical alignment of the system. If a battery error occurs these values are lost and must be reintroduced. Reintroduction is effected using the correct θ and the correct 2θ commands, which must only be used with extreme caution. When producing a normal diffractogram the integrity of the 2:1 relationship between the shafts is vital, or the detected peaks will be distorted. The correct angle readout, as obtained from the alignment with the single and double knife edges should be maintained (ZERO2T and ZEROT). The correct θ and 2θ commands should not be used when this relationship has been accurately defined.

CAN This command is disabled when operating the system with a PW1747, and the error message displayed on entry of the command is:

** 4 ILLEGAL COMMAND

8.6 PW1748 SECOND DETECTOR

A second detector option is provided for special applications. This is found in a fixed position on a modified diffractometer. Typical applications of this option are monitoring scattered radiation, fluorescent radiation, or specific diffraction lines. Designation of this added detection channel is by addition of an "F" to relevant commands.



LWLF 1 Set the lower level to "1%". The value "1%" must be an integer in the range zero to 99 inclusive.

UPLF 1 Set the upper level to "1%". The value "1%" must be an integer. Selection of an upper level of zero causes the level to be set quite high, at approximately 150%.

RINF Recall the intensity measured on the second detector, channel "2", to the accumulator. The intensity is stored in a separate register in the same manner as the intensity measured with the first detector. Similarly the value held in this register is lost as soon as another measurement starts.

RCTF Recall the counts measured on channel "2" to the accumulator. The measurement of counts from the second detector is stored in a separate register, and maintained until another count is taken, in the same manner as the first detector.

RFSF iEj The recorder full scale command is used to set the recorder to record intensities across its full scale. When the recorder is operating it indicates the actual intensity like a meter, thus acting as a ratemeter. The record would not be very useful if travel only occurred across a limited segment of the scale. For this reason the full scale deflection can be adapted to the known (or expected) maximum intensity value. The range of possible settings is from 100 (1E2) to 500,000 (5E5) counts per second for full scale. Ratemeter full scale can be any of the following, (1,2,5)E(2,3,4,5).

PHDF This command is used to produce a pulse height distribution curve for the second channel. It operates in precisely the same manner as the pulse height distribution curve facility on the first channel.

8.7 PM4202 CASSETTE RECORDER

The PM4202 cassette recorder option is provided to allow storage of user made programs. As detailed previously the PW1710 incorporates a four kbyte random access memory to hold programs. A certain amount of this memory is used to store calibration constants. Registers also use some of this space. Approximately 3,500 bytes remain for programs. The cassette recorder allows storage of these programs, meaning that different groups of programs can be stored on separate cassettes and reloaded onto the system when required.

The recorder is connected to the system via the second connector on a PW1712 dual serial interface. This connector can also be used for a printer. It is important to remember that selective storage of programs on cassette, or selective reloading, is not possible. This means that when the programs stored on a cassette are loaded onto the system all the programs held in memory are overwritten. The commands used when the PM4202 option is fitted are detailed in the following paragraphs. Although it is necessary to select dual output when connecting a printer the cassette recorder has a special two-way communication system meaning that dual output selection is not necessary.

WME This command is used to write the programs currently held in memory out to a cassette.

RME This command is used to read all the programs stored on cassette into the memory. It is very important to remember that all the programs held on the system before entry of this command will be overwritten by those stored on cassette.

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TO BE ISSUED LATER

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MA22909